Physical Annealing During Oven-Ageing of CARILON Polymers

J. M. Machado, P. B. Himmelfarb, S. C. Tang, R. M. Irwin, J. S. Grebowicz, C. A. McDaniel, M. P. Williams

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Westhollow Research Center
Houston, Texas

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ABSTRACT

A complementary set of analytical techniques indicate that CARILON EP polymers experience an increase in their degree of crystallinity by 5-10% in the first stages of oven-ageing. This physical annealing effect appears to exert a profound influence upon mechanical properties and must be taken into account when interpreting oven-ageing results.

TECHNICAL INFORMATION RECORD WRC

PHYSICAL ANNEALING DURING OVEN-AGING OF CARILON POLYMER

by

J. M. Machado, P. B. Himmelfarb, S. C. Tang, R. I. Irwin,
J. S. Grebowicz, C. A. McDaniel and M. P. Williams

INTRODUCTION

It is widely recognized that CARILON polymers undergo chemical oxidative degradation when exposed to air at elevated temperatures for extended periods of time [1,2]. Accelerated ageing tests are performed to measure the effect of these degradative reactions upon various relevant properties and to predict material longevity at contemplated use temperatures. The intelligent interpretation and application of such ageing data reqire the appropriate selection of properties to monitor and criteria for failure. Some knowledge of the chemical and physical processes which give rise to property changes is also very desirable.

In the present contribution we will provide evidence derived from a diversity of analytical techniques which demonstrates that that significant physical changes (annealing) preceed chemical changes during oven-ageing experiments with CARILON polymers. Depending upon the property under evaluation and the criterion for failure, these physical changes can strongly influence oven life.

EXPERIMENTAL

CARILON EP polymers are copolymers of ethylene, propylene, and carbon monoxide. The material used in this study was CARILON EP polymer batch 89/035 having a reported LVN and melting point of 1.8 dl/g and 223C respectively. Standard test specimens were prepared by injection molding of dried pellets using a Kraus-Maffei 100 ton molding machine. Specimens were stored over dessicant and tested "dry as molded". Specimens for ageing studies were placed in a forced air oven maintained at 125C. Samples were removed periodically and tested in the "dry as aged" state.

Samples for analysis were also stored over dessicant prior to testing. Samples for various analyses were prepared from injection molded tensile bars by the microtoming of appropriately thin sections. In most cases, samples were taken from the "bulk" or central core of the molded part and from the "skin" or outermost region of the part. Analytical specimens were prepared from unaged samples and from samples aged for 24 hours at 125C. Additional analytical procedural information is provided in the appendices.

SUMMARY OF RESULTS

Mechanical Properties

The samples experience a very large change in mechanical properties within the first three hours of ageing. This is much too short a period of time for any appreciable atmospheric oxygen to diffuse into the sample, thus raising the suspicion that the effect is not chemical but physical in nature. (Recent results show that similar initial property changes also occur while ageing in an inert atmosphere.) [3]

Within 24 hours the samples lose more than 50% of their original tensile elongation and notched Izod values, which fulfills the criterea for sample failure according to Underwriters Laboratory specifications [4]. Accompanying this change is a significant increase in the tensile yield stress and a modest increase in tensile yield strain. An increase in yield stress upon ageing is most readily explained by an increase in crystallinity. Based on the fact that yield stress is roughly proportional to crystallinity, we estimate that the observed increase in yield stress upon ageing for 24 hours corresponds to an increase in the absolute degree of crystallinity by 5-8%.

X-Ray Diffraction

WAXD studies suggest that the bulk material is more crystalline than the skin material, but that both bulk and skin materials experience a significant increase in crystallinity upon ageing for 24 hours. The estimated increase in absolute crystallinity in the bulk sample during ageing is 8%.

Thermal Analysis

DSC results are in agreement with the scattering data, showing an increase in crystallinity in both the bulk and skin regions upon ageing. Qantitation of the enthalpies of melting from the bulk region indicate an increase in the degree of crystallinity by 6% upon ageing for 24 hours.

Careful inspection of the melting peaks also indicates an increase in the degree of crystalline perfection upon ageing.

Optical Microscopy and Birefringence

Microscopy of cross-sections of unaged and aged specimens demonstrates that both materials possess a spherulitic core and an apparently oriented skin region which is several hundred microns in total thickness and which is itself composed of several morphologically distinct layers. No obvious change in the appearance of these cross-sections occured upon ageing.

Birefringence measurements performed at various points in these cross-sections indicated that there was an overall decrease in birefringence upon ageing for 24 hours, suggesting a relaxation of "molded-in" orientation.

Infra-red Spectroscopy

FTIR did not reveal any evidence of degradation or chemical change during ageing for 24 hours, either in the bulk or the skin regions. However, examination of known polyketone "crystallinity bands" indicated an increase in the degree of crystallinity in both regions upon ageing.

DISCUSSION

Clearly, there is ample agreement between the various methods to conclude that CARILON polymers experience an increase in total crystallinity and probably in crystalline perfection within the first few hours of oven-ageing at 125C. This should not be surprising since the ageing experiments are carried out about midway between Tg and Tm where the driving force for secondary crystallization reaches a maximum, owing to the combined contributions of super-cooling and segmental mobility. This annealing effect is entirely physical in nature and occurs in addition to (and largely preceeding) chemical degradation.

It seems very likey that the initial changes in properties during oven-ageing experiments are associated with annealing effects. It should be emphasized however that other factors may also contribute to this change. One factor which cannot be entirely ruled out is degradation by oxygen dissolved in the molded sample. Another possibility is the further removal of absorbed water under the conditions of ageing [5]. Although the samples were tested and aged in the "dry as molded" state, they may still contain some moisture.

The results indicate that physical and morphological factors must be taken into account when interpreting property changes in CARILON polymers subject to elevated temperatures. Further investigation in this area is needed.

CONCLUSIONS

CARILON polymers experience an increase in their degree of crystallinity of 5-10% in the first stages of oven-ageing. This physical annealing effect can have profound influence upon mechanical properties.

REFERENCES

1. R.Q. Kluttz, WRC-TPR 47-88

2. J.L.M. Syrier, KSLA-AMGR 89-196

3. R.Q. Kluttz and A.A. Broekhuis, personal communications

4. UL specifies that the time at which a 50% change in the initial value of any property is observed is the time to failure.

5. W.P Gergen, personal communication.

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APPENDIX 1

Appendix 1.

Mechanical Properties

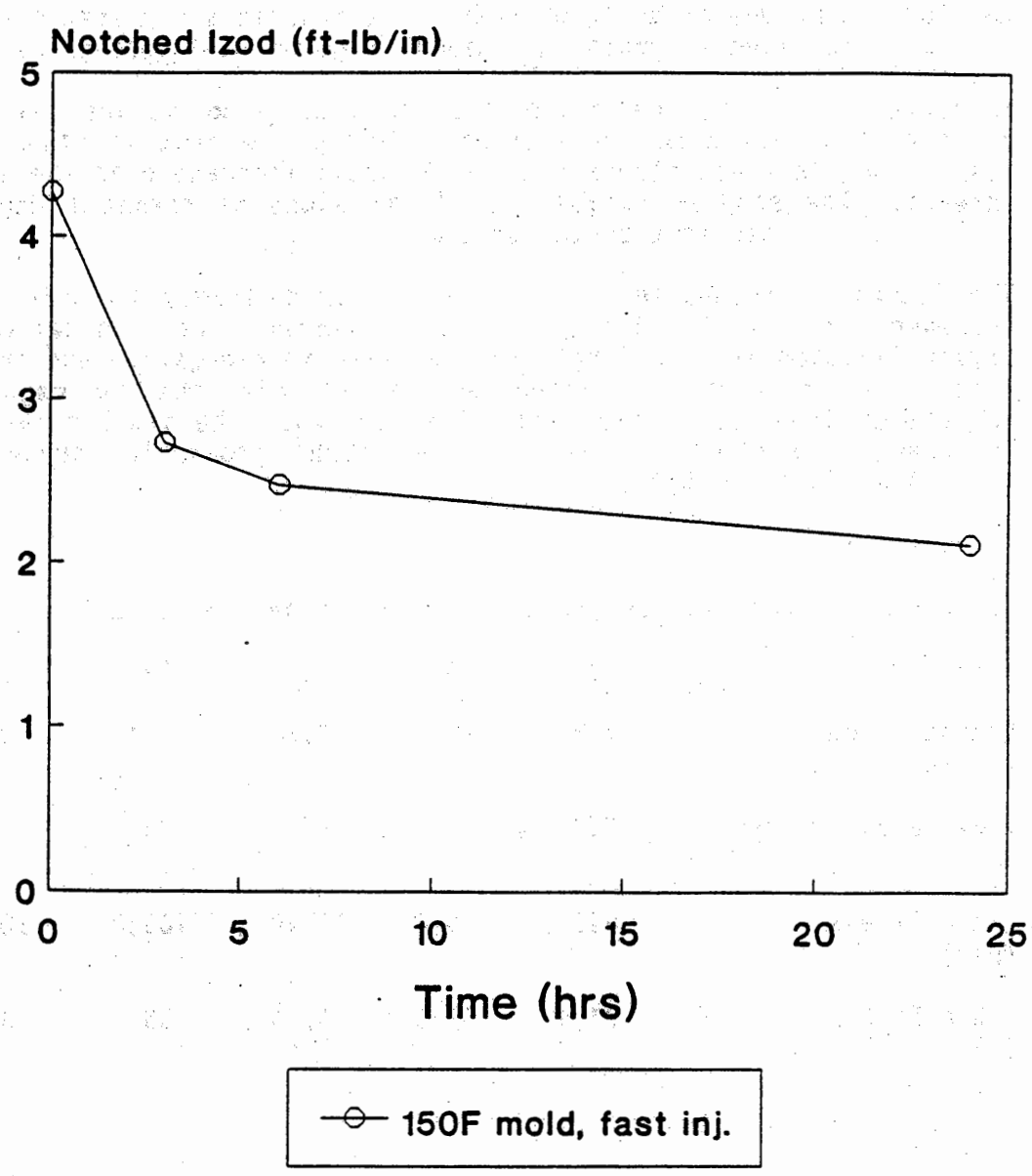
Injection molded samples of CARILON polymer 89/035 were placed in a forced air oven maintained within one degree of 125C. Samples were withdrawn periodically and subject to tensile and notched Izod tests. All Izod bars were notched prior to ageing. Properties were tested in the "dry as molded or as aged" state. The results are presented in Table A1-1 and are given in graphical form in Figures A1-1 through A1-3.

Essentially, it may be noted that notched Izod values and tensile elongation values drop precipitously during the initial stages of the test. Tensile yield stress and yield strain increase over the same time interval. The samples exhibit no visual signs of chemical degradation (yellowing) within this period of time.

The increase in tensile yield stress is most likely to arise from an increase in crystallinity during ageing (a so-called cold crystallization or annealing). The decrease in elongation and in notched Izod could arise from an increased crystallinity but the magnitude of the effect is surprisingly large. Nevertheless, the similar ageing time dependance displayed by all four mechanical properties suggests that they have similar origins.

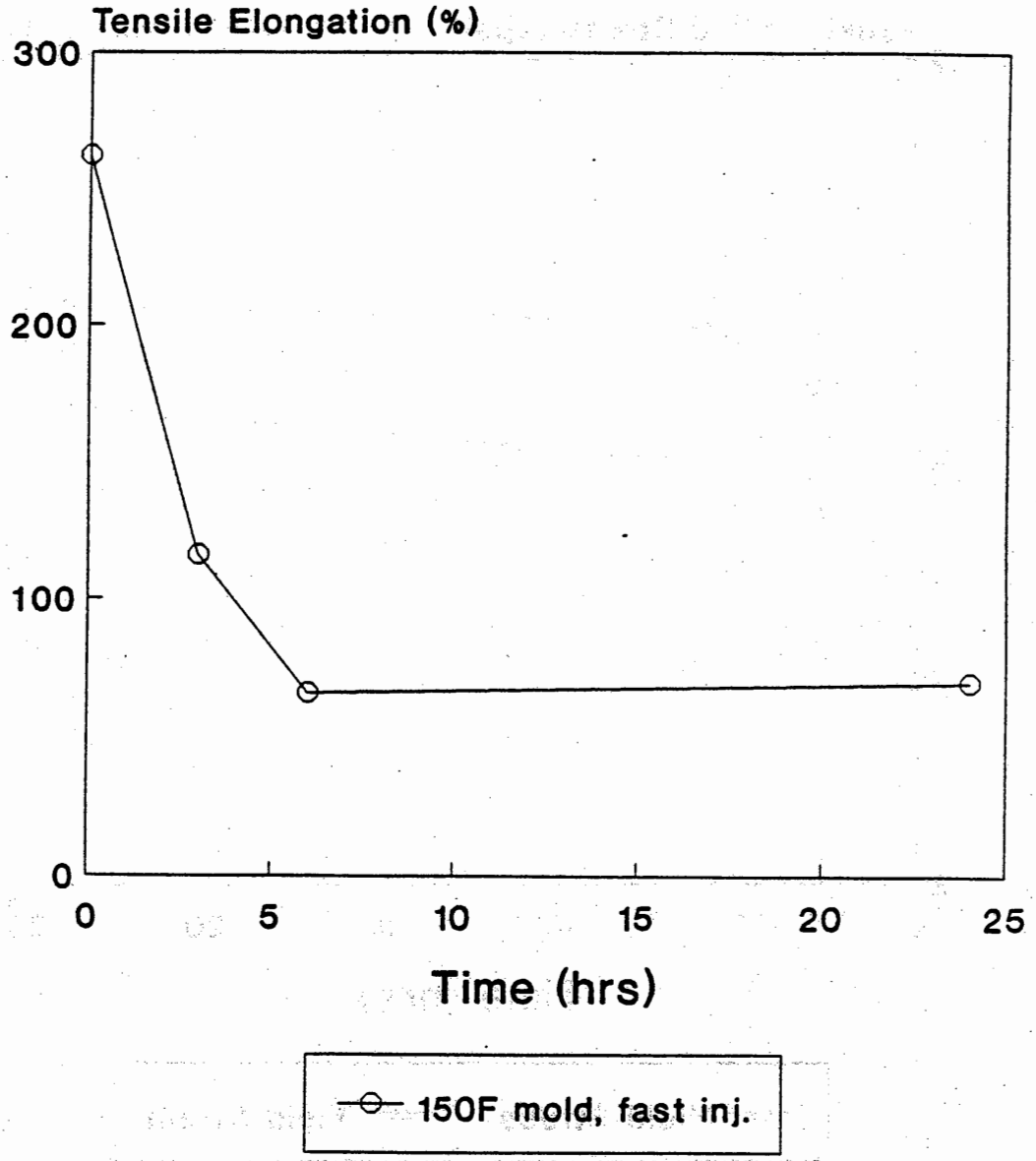
Table A1-1. Mechanical properties during ven ageing at 125C.

Time (hrs)	0	3	6	24	96
Notched Izod (ft-lb/in)	4.3	2.7	2.5	2.1	0.9
Tensile Elongation (%)	262	117	66	70	45
Yield Stress (psi)	8980	9910	10040	10320	10720
Yield Strain (%)	31.4	33.0	33.4	33.5	33.5



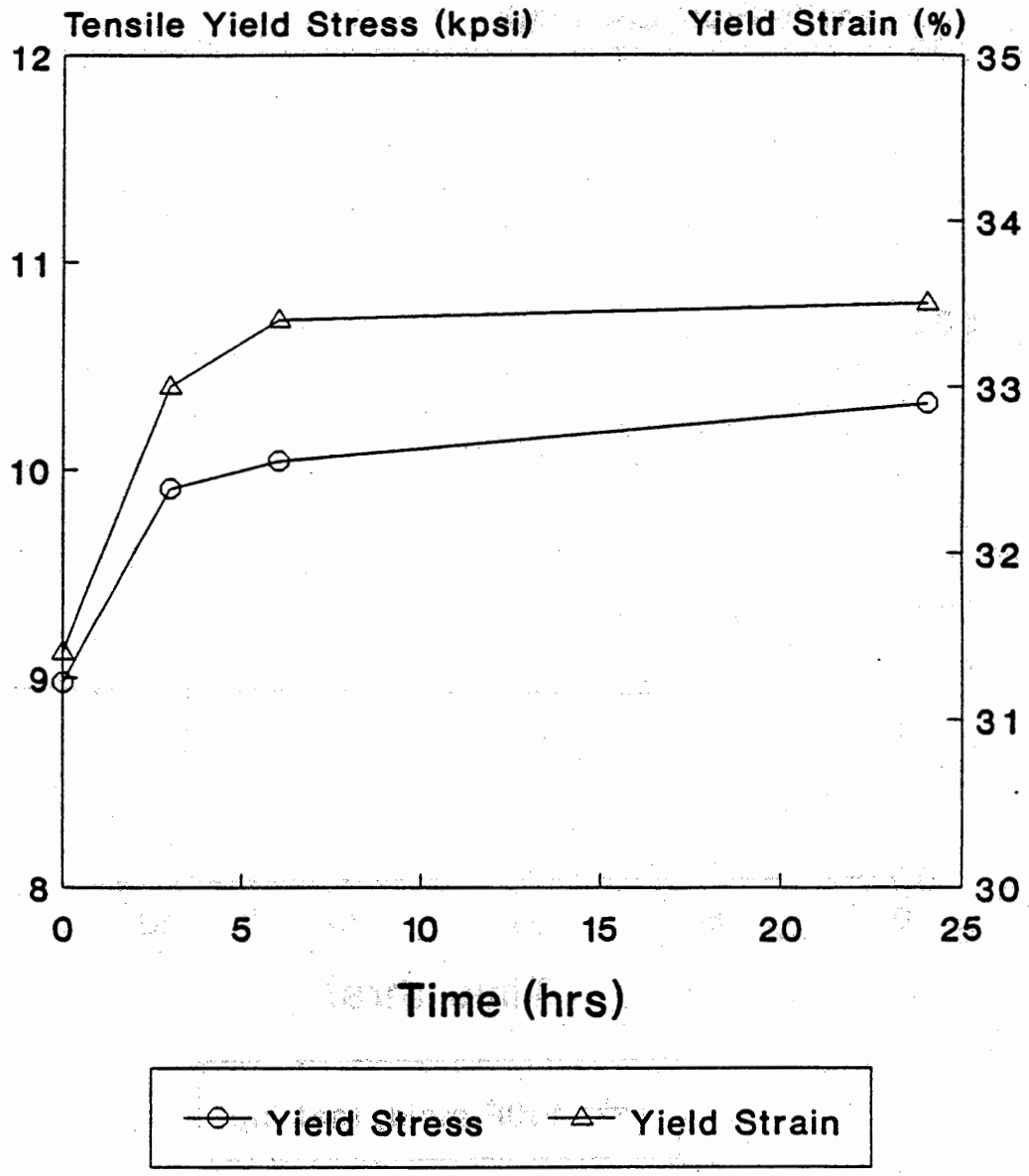
CARILON Polymer 89/035
Aged at 125C; forced air

Figure A1-1



CARILON Polymer 89/035
Aged at 125C; forced air

Figure A1-2



CARILON Polymer 89/035
Aged at 125C; forced air

Figure A1-3

APPENDIX 2

APPENDIX 2

January 19, 1990

TO: J. M. MACHADO

FROM: M. P. WILLIAMS

SUBJECT: X-RAY DIFFRACTION RESULTS OF CARILON SAMPLES (WRC 250521)

The X-Ray Diffraction lab was given several small pieces of both the MG59A - Control and the MG59A - 24 hr/125 C Carilon samples taken from the skin and the bulk portions. Pieces of each of the samples were placed into four separate x-ray sample holders and all were run on the diffractometer. After looking at each scan and performing the calculations, it was observed that there was a definite increase in crystallinity in the skin portions from the control to the treated sample, but the bulk portions were not as definitive.

It had been noticed that there are grains in each sample's pieces which raised the question of whether it made any difference in the results if the x-ray beam scanned across the piece vertically or horizontally. Therefore, each sample was rerun being careful to place the pieces all vertically in one sample holder and all horizontally in a second holder. After looking at each scan and performing the calculations the following results were obtained:

SAMPLE	RESULTS		
MA59A Control Skin	Vertical	41.72%	average
	Horizontal	39.38%	40.6%
MG59A Control Bulk	Vertical	55.21%	average
	Horizontal	46.26%	50.7%
MG59A 24hr/125 C Skin	Vertical	53.58%	average
	Horizontal	54.45%	54.0%
MG59A 24hr/125 C Bulk	Vertical	52.82%	average
	Horizontal	65.04%	58.9%

A couple of observations can be made from the data. The first observation being that it didn't seem to matter with the skin portions whether or not the pieces were positioned vertically or horizontally, but the bulk portions did seem to show an orientation effect; unfortunately, it was not the same on both samples. The second observation was the definite increase in crystallinity for both the skin and the bulk portions from the control to the treated sample. It should be noted that the crystallinity was calculated by measuring the total area then measuring the area of the amorphous portion

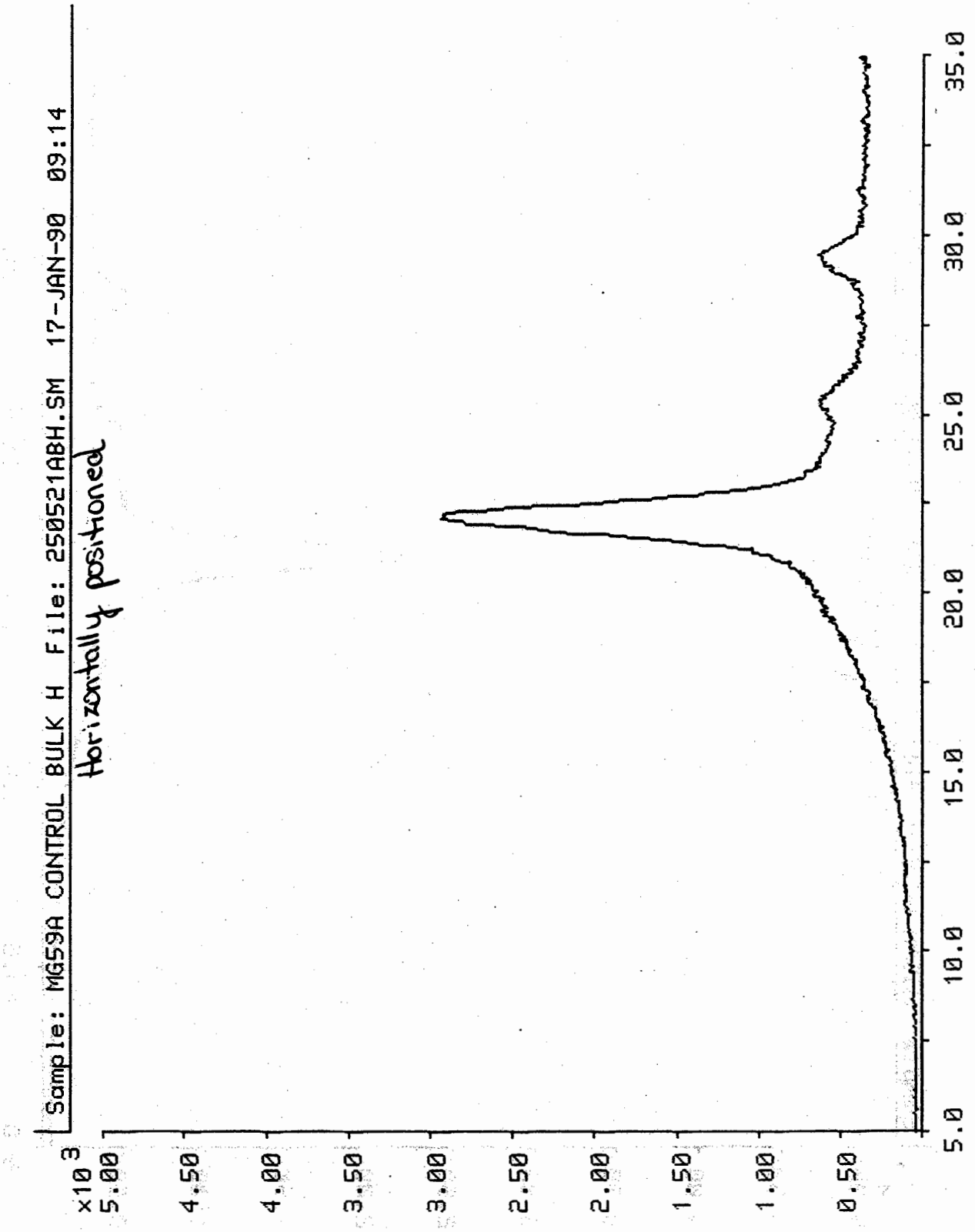
after which a simple division to determine the percent of the amorphous content was made and subtracted from the total to obtain the percent crystallinity.

Enclosed with the report are copies of the scans taken both vertically and horizontally of each skin and bulk portions of the two samples.

M. P. Williams

M. P. Williams

cc: R. D. Cates
S. C. Tang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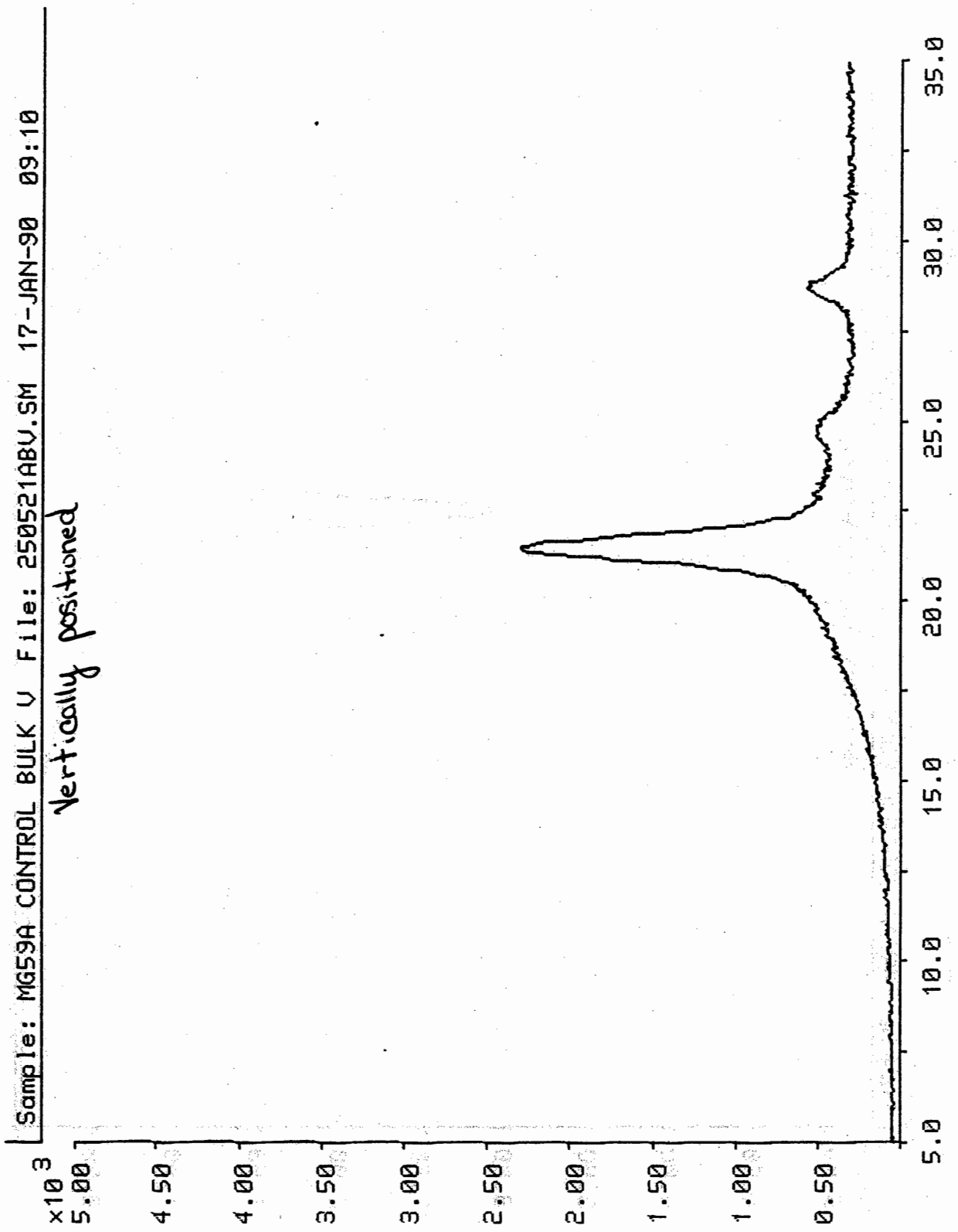
5.00
4.50
4.00
3.50
3.00
2.50
2.00
1.50
1.00
0.50
5.0
10.0
15.0
20.0
25.0
30.0
35.0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4.50
4.00
3.50
3.00
2.50
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5.0
10.0
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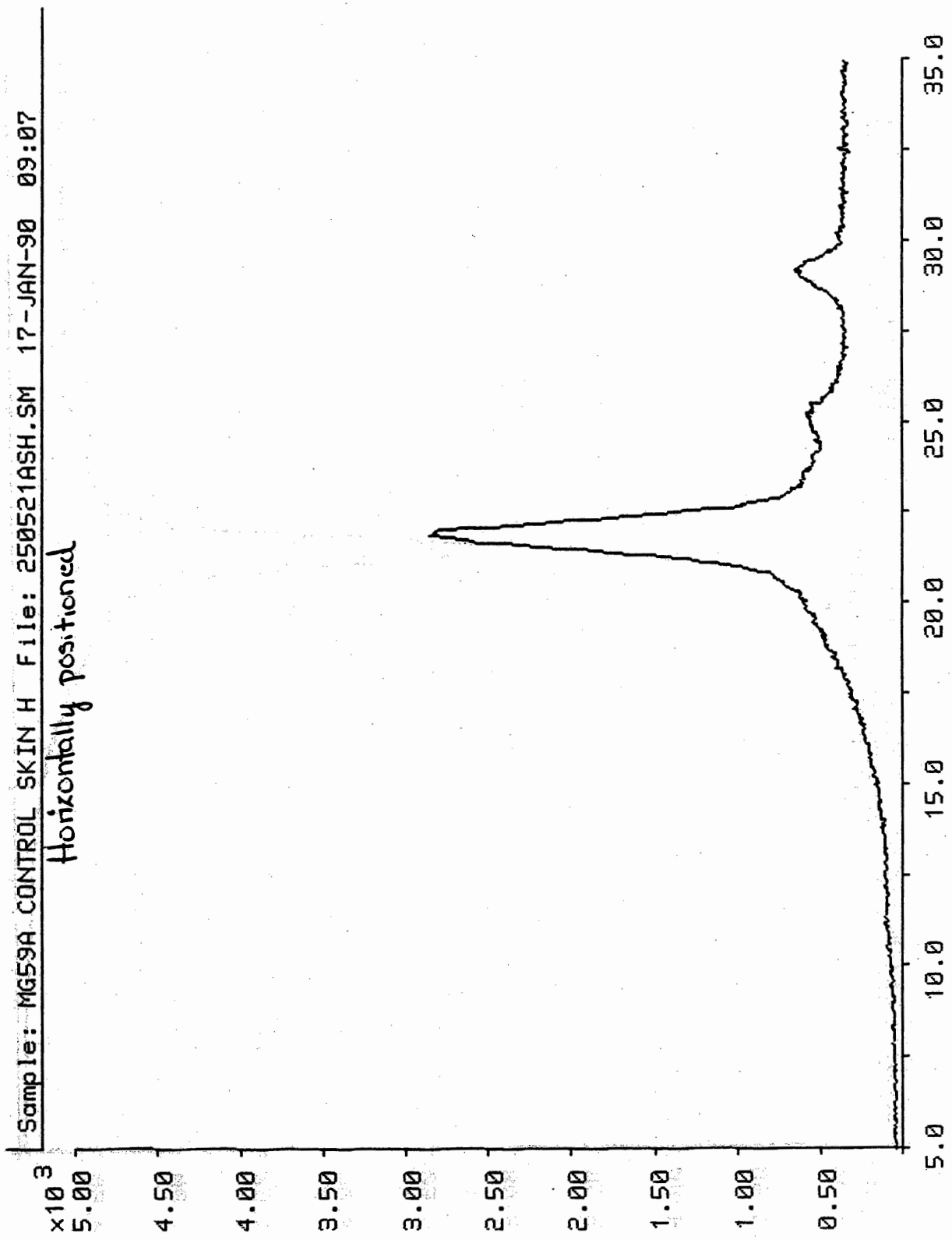
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4.00
3.50
3.00
2.50
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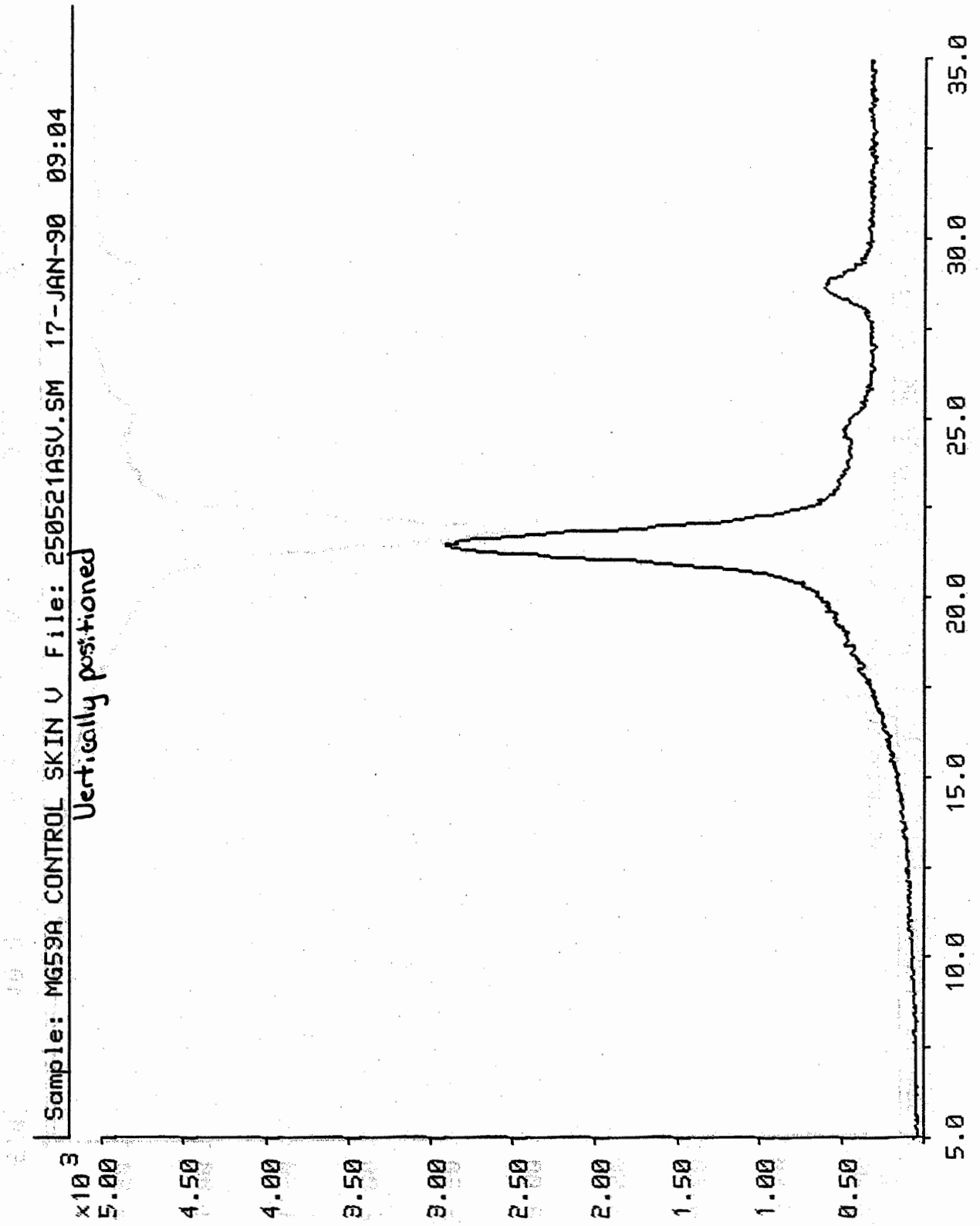
1.50
1.00
0.50
5.0
10.0
15.0
20.0
25.0
30.0
35.0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5.00
4.50
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3.00
2.50
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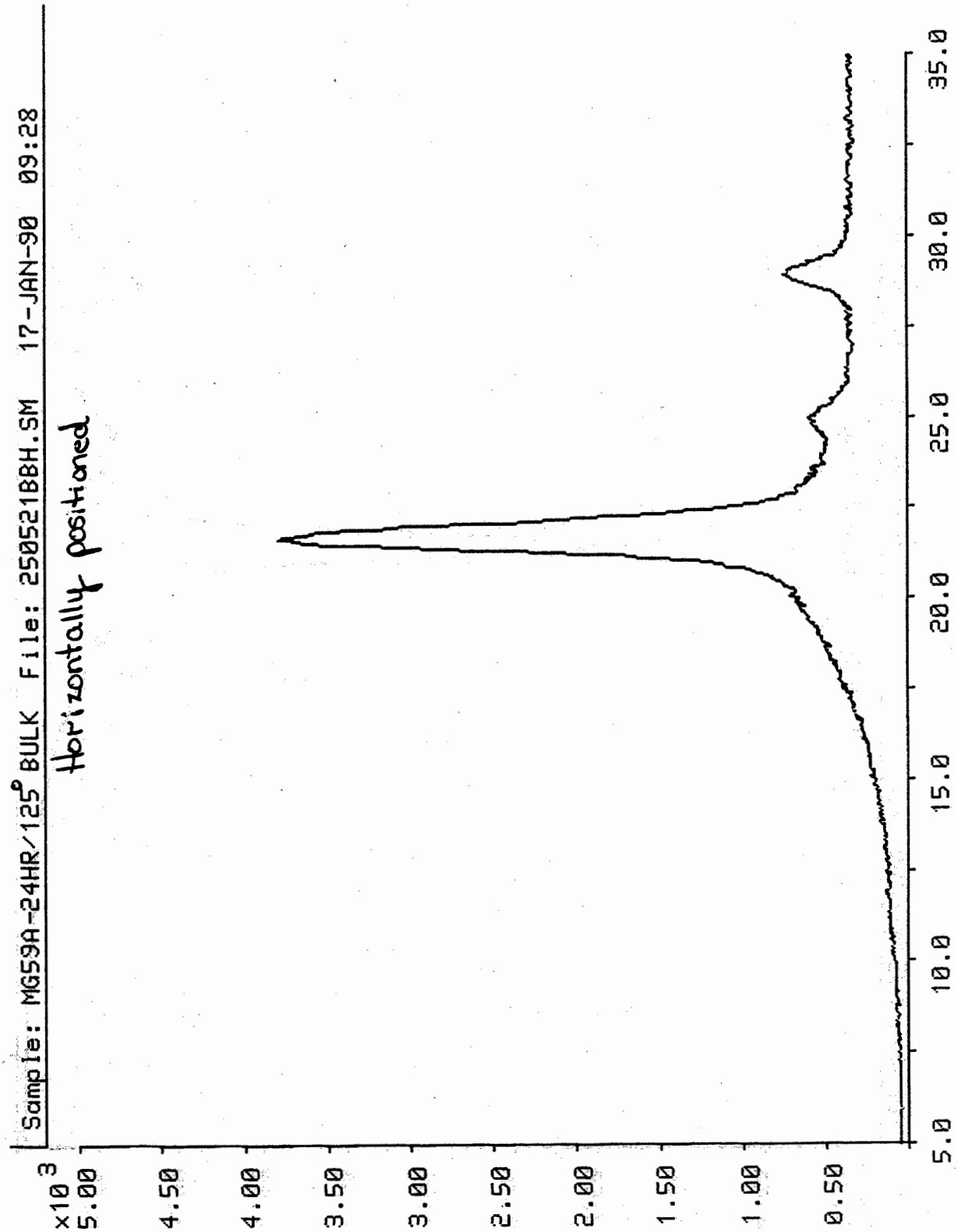
1.50
1.00
0.50
5.0
10.0
15.0
20.0
25.0
30.0
35.0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3.50
3.00
2.50
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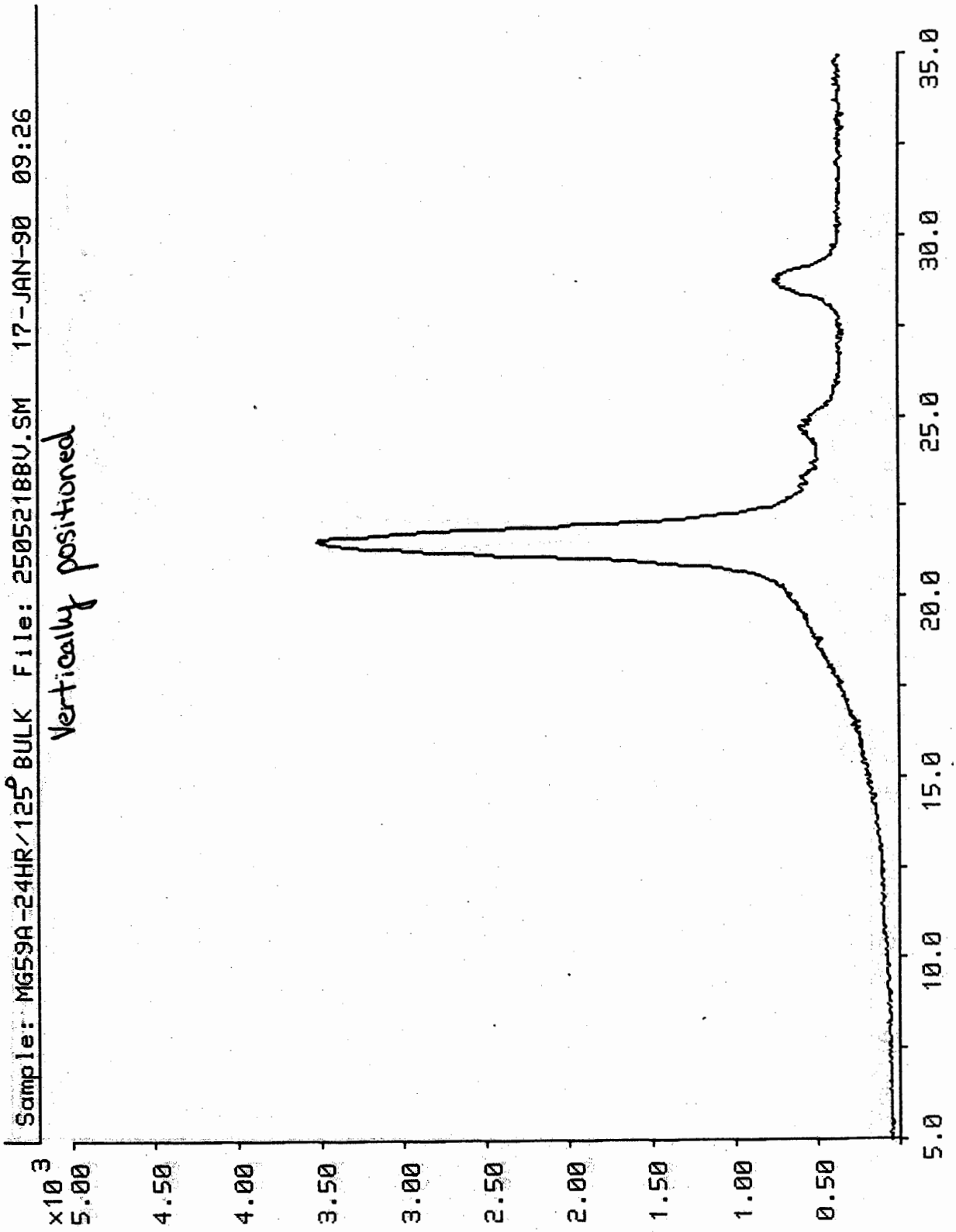
15.0
20.0
25.0
30.0
35.0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4.50
4.00
3.50
3.00
2.50
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1.00
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5.0
10.0
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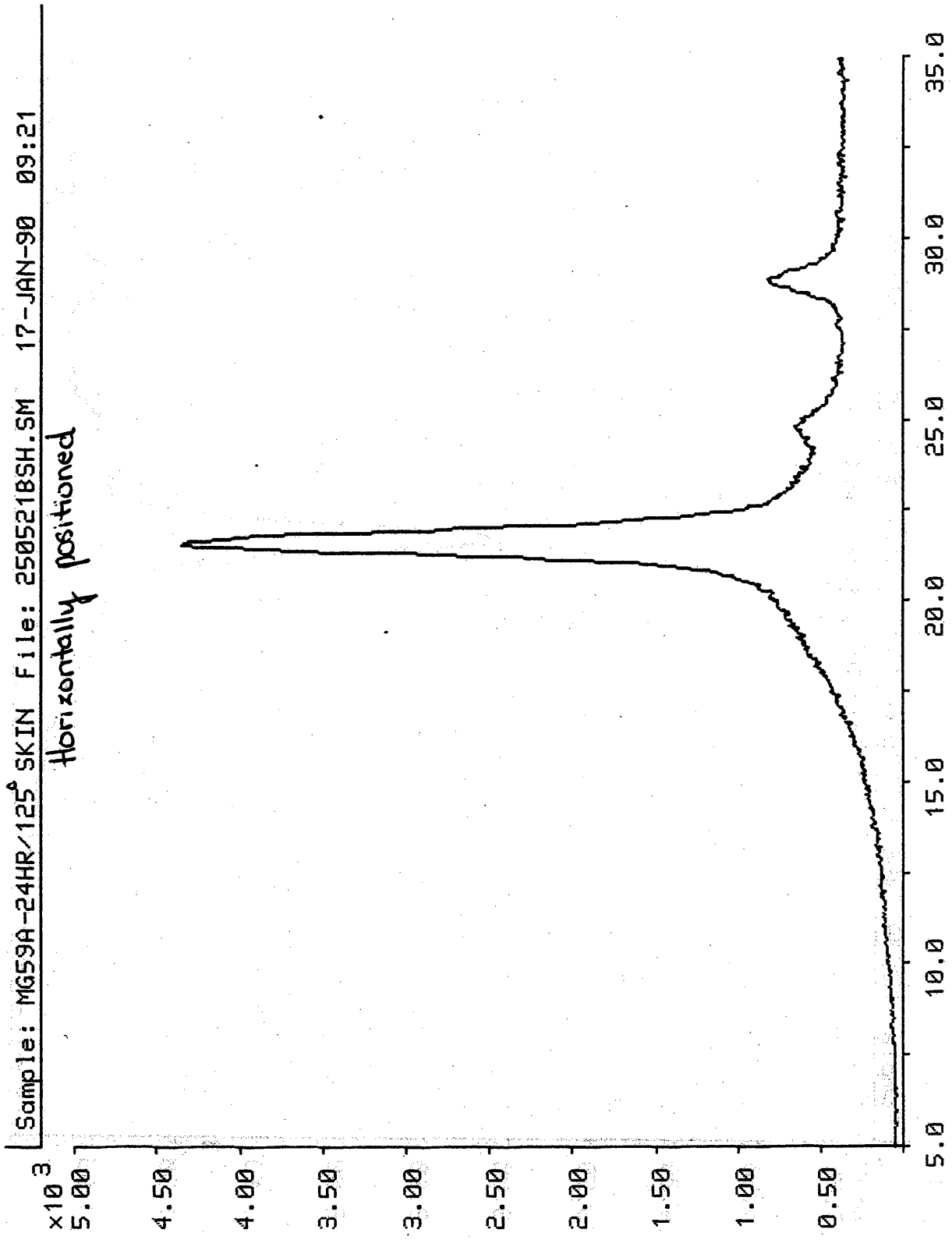
30.0
35.0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4.00
3.50
3.00
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10.0
15.0
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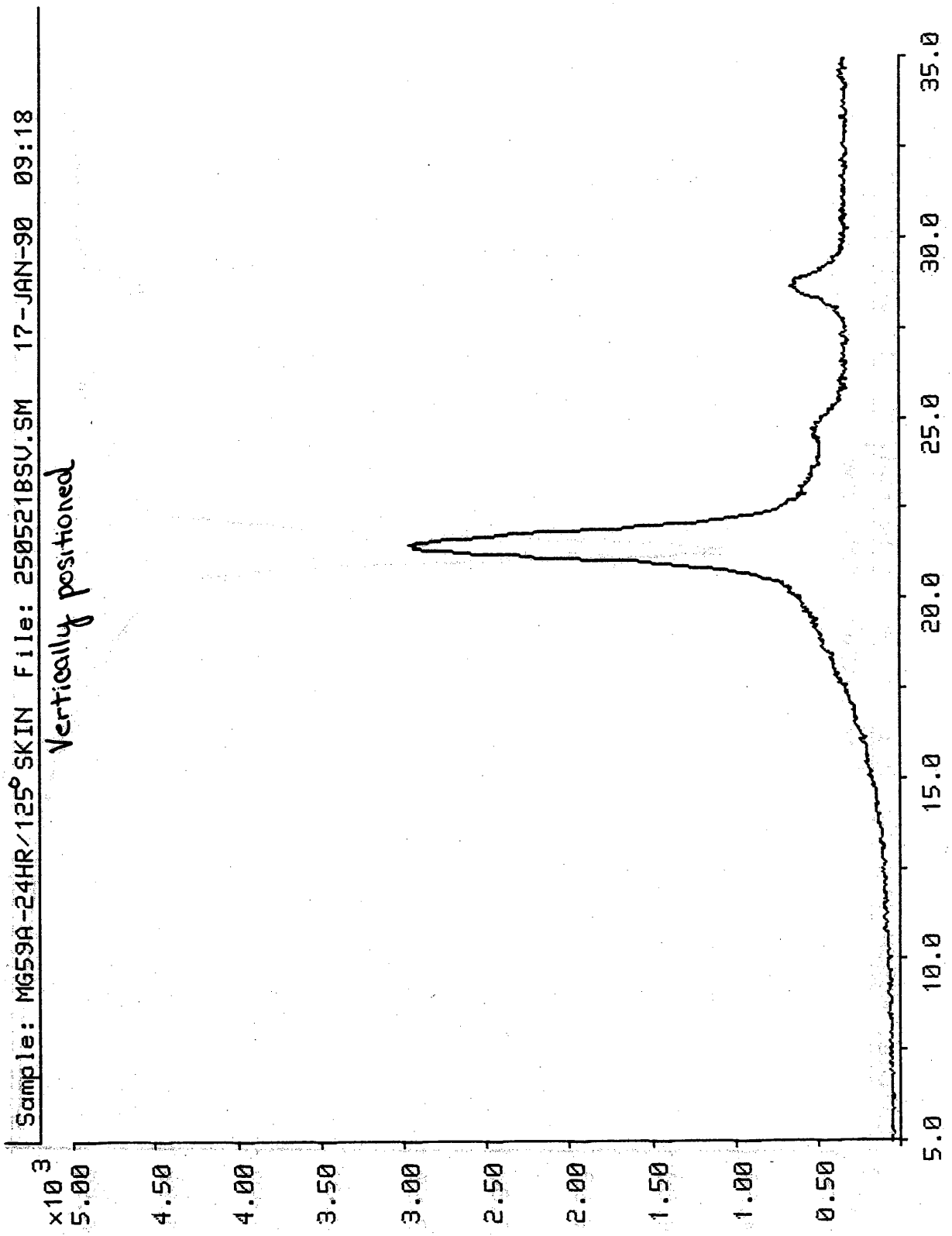
25.0
30.0
35.0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2.00
1.50
1.00
0.50
5.0
10.0
15.0
20.0
25.0
30.0
35.0

APPENDIX 3

APPENDIX 3

Thermal Analysis

Thermal analysis was performed with Perkin-Elmer DSC in the temperature range of -40-285°C. Samples of bulk, skin-line and skin surface for control and heat treated materials were submitted. The analysis was focused on melting behavior. Some observations were made regarding glass transition. A heat of melting 55 cal/g (230.12 J/g) value was used.

Results

Figure 1 and 2 show DSC traces respectively for control and annealed samples. Two major thermal events were recorded: glass transition between approximately 0-60°C and melting at about 140-220°C. In both cases, the melting peaks are structured showing smaller peaks or shoulders either on the low or high temperature side of the main peak. The main qualitative difference in DSC behavior between the two materials is the presence of a broad annealing peak above 130°C in thermally treated sample, which simultaneously marks the beginning of melting. Numerical data concerning this process are in Table 1. In control material the bulk portion shows slightly higher crystallinity: 36% as opposed to 34% for the outside regions. Temperatures of the main peak shift from 218°C (bulk and skin like) to 221 (skin surface).

In annealed samples peak temperature changed very little. Main peaks broaden so what was observed as a high temperature peak in control material now became a shoulder (on high temperature side). On low temperature side beginning of melting occurs 10-20°C higher than annealing temperature, which is a typical result of annealing of semicrystalline polymers. This is an indication that some perfection of crystals takes place. Overall crystallinity increased by 6, 5 and 3% respectively for bulk, skin-like and skin surface.

In all cases broad, two step glass transition was recorded between about -5-65°C with the major step mid-points at about 5 and 55°C. The typical illustration of this observation is given in Figure 3.

Conclusion

Annealing of Carilon leads towards our overall increase of crystallinity and crystal perfection. The most dramatically of these are shown in the bulk part of the sample.

Table 1 Melting parameters of Carilon samples.

	Control			Annealed 125°C, 24 hrs		
	Tm, °C	ΔH, J/g	Cryst,%	Tm, °C	ΔH, J/g	Cryst,%
Bulk	218(m) 223	83	36	218(m)hts 130(b)	96	42
Skin like	217(m) 222	79	34	217(m)hts 135(b)	90	39
Skin surface	217 221(m)	79	34	213,219, 223(m) 140(b)	82	36

(m) main peak
hts high temperature shoulder
(b) begining of transition

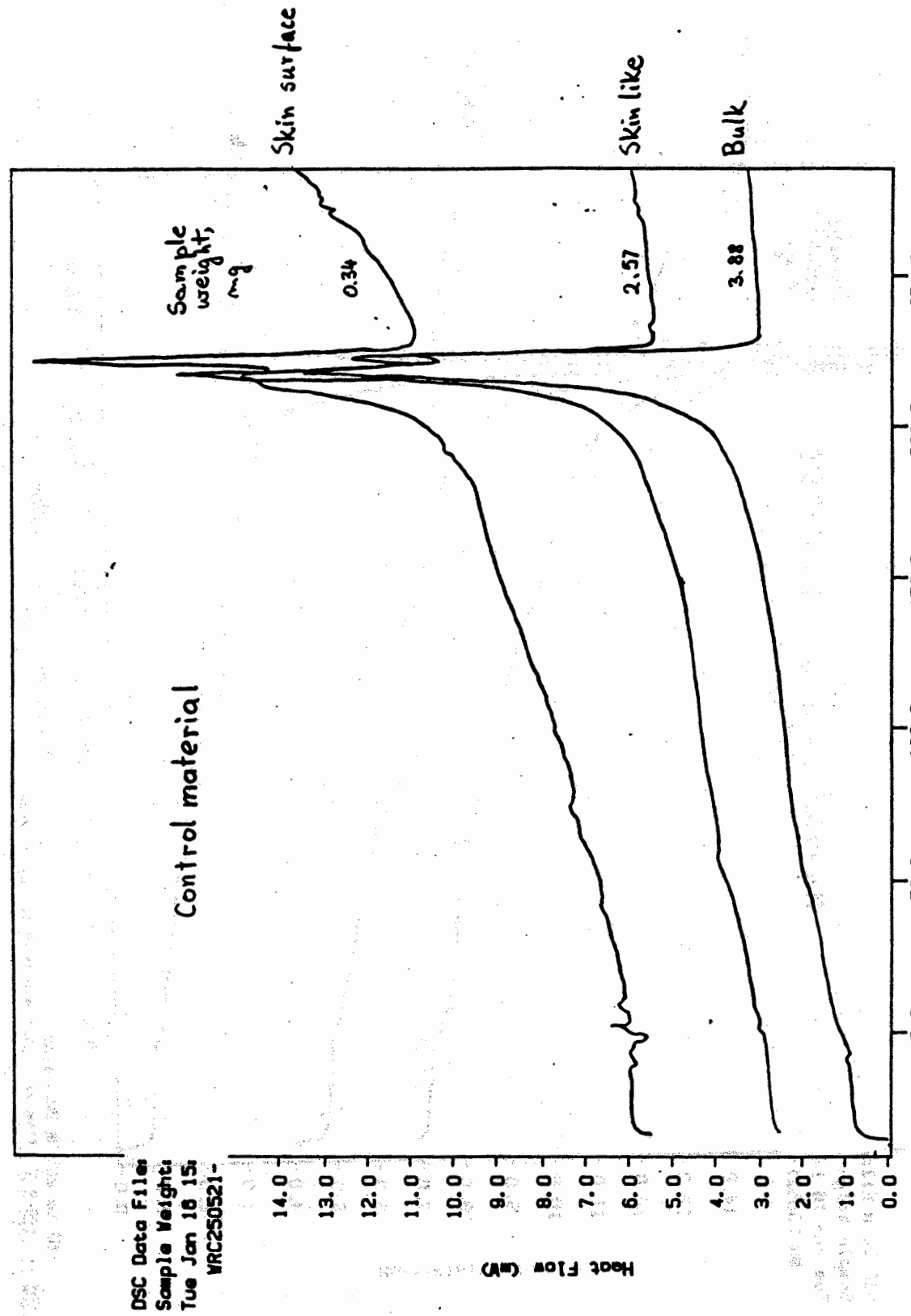

Fig 1. DSC traces of control material

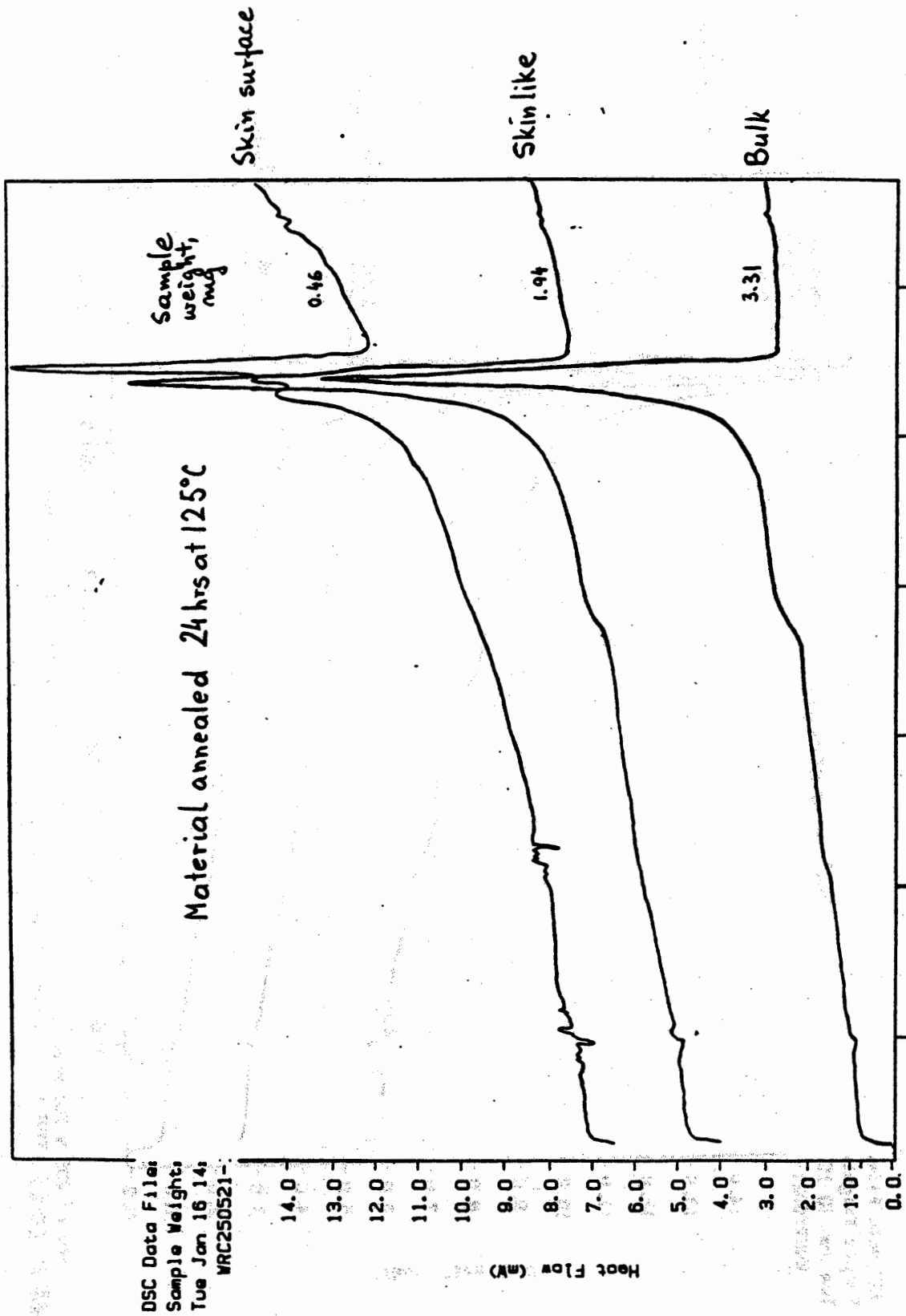

Fig 2. DSC traces of annealed material

DSC Data File: 11604
Sample Weight: 3.310 mg
Tue Jan 16 15:53:14 1990
WRC250521-2A MG59A 24hrs @ 125 BULK

PERKIN-ELMER
7 Series Thermal Analysis System

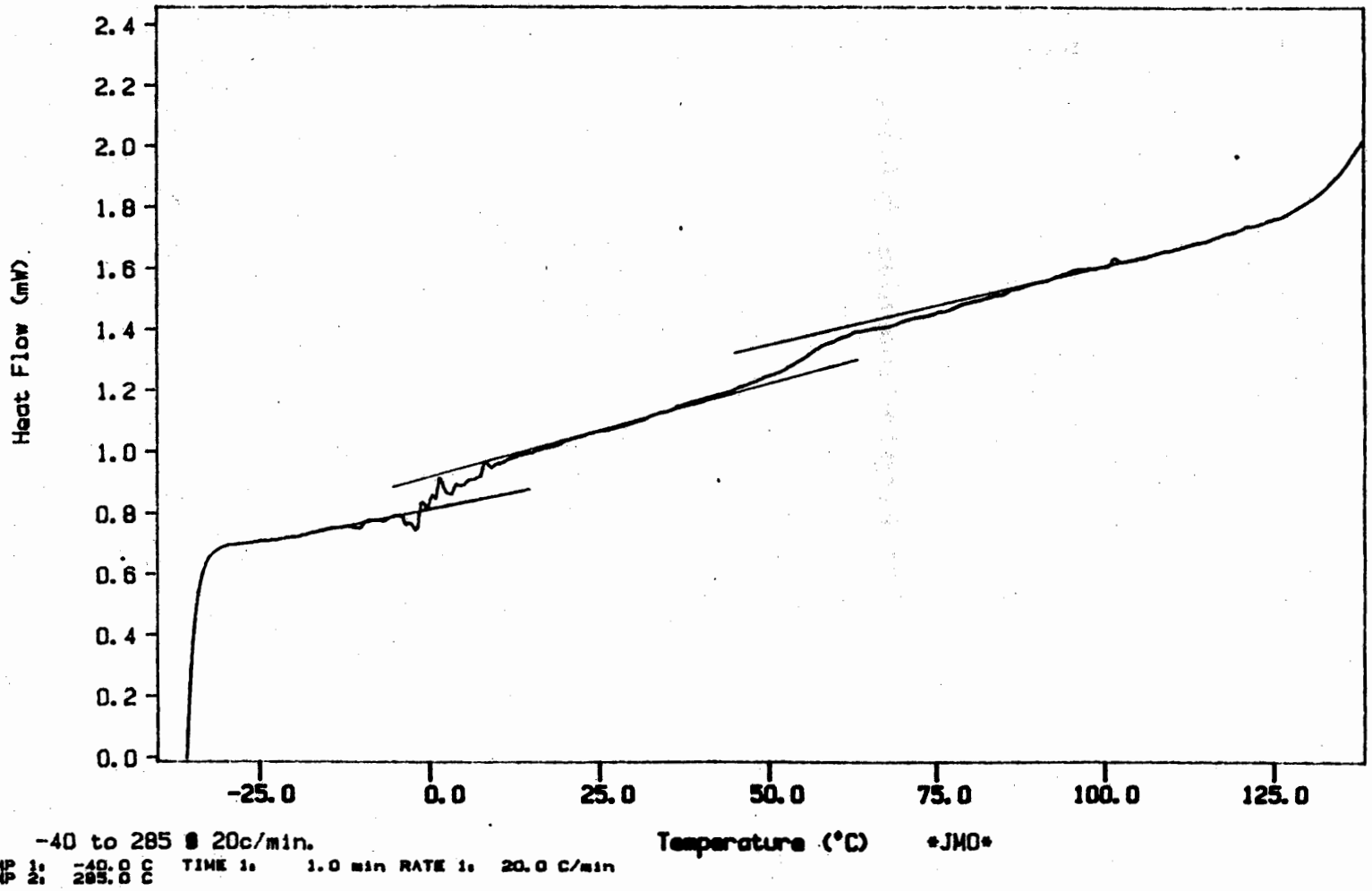

Fig. 3. Typical illustration of two step glass transition in Caviton

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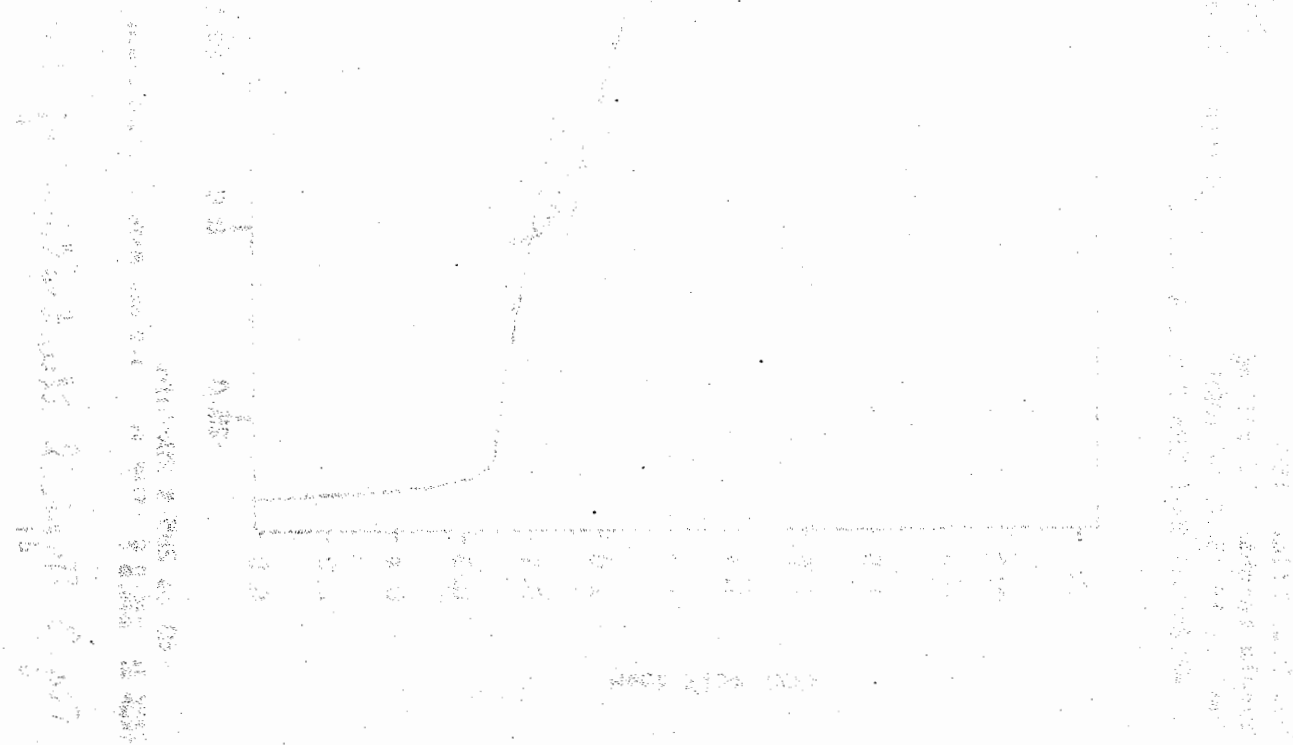


APPENDIX 4

APPENDIX 4

CAM/WR250521.DOC

SKIN THICKNESS AND BIREFRINGENCE IN CARILON POLYMER

CUSTOMER: Machado, J.
REQUEST: WRC #250521
ANALYST: C. A. McDaniel
DATE: January 5, 1990

OBJECTIVE: Determine the skin thickness and birefringence in heat treated Carilon Polymers. Two samples submitted
1) referance - MG-59A Control
2) heat treated - MG-59A 24Hr/125C

PROCEDURE: The birefringence in Carilon Polymers was determined using the Leitz Orthoplan Polarizing Microscope and the Leitz Tilting Compensator. The samples were prepared for analysis by microtoming 4um sections using a Leitz Rotary Microtome across the center of the tensile bar. The samples were cut at room temperature.

The following steps were used to achieve the birefringance measurement:

1) The 4um thick section of Carilon was mounted on a microscope slide using Aquamount and a No. 1 1/2 cover slip.
2) The microscope slide was placed on the stage of the microscope and rotated to an extinction position in crossed polars.
3) Turning the sample 45 degrees to the left, the compensator was inserted into the slot and two tilting angles were measured.
4) Magnification used to achieve birefringence measurements was 630x which covered an area approximately 135 um by 173 um.
5) Three sets of measurements were made at eleven areas along the section. The average of these values is reported below for birefringance.

Birefringence calculations were made using the following equation:

Retardation(nm) = 2i(two tilting angles)
Thickness(um)

Birefringence=
.Retardation/(thickness(um)*1000(nm/

BIREFRINGENCE

AREA #	MG-59A CONTROL	MG-59A 24HR/120C
1	1.9 X 10-1	6.8 X 10-2
2	3.9 X 10-1	3.3 X 10-1
3	4.3 X 10-1	3.8 X 10-1
4	3.4 X 10-1	2.6 X 10-1
5	3.9 X 10-1	3.6 X 10-1
6	1.8 X 10-1	1.8 X 10-1
7	4.0 X 10-1	3.8 X 10-1
8	3.4 X 10-1	3.0 X 10-1
9	4.2 X 10-1	3.5 X 10-1
10	3.6 X 10-1	3.3 X 10-1
11	2.4 X 10-1	9.2 X 10-2

See graph attached.

.Skin thickness was determined using crossed polars at 100x magnification. A 100x montage was also made in crossed polars of each sample to show the structure of the whole section. Different bands were observed in the micrographs which are labeled Area 1 through Area 11. Birefringence was measured in these same eleven areas.

CONCLUSIONS

1) Eleven distinct bands were observed from edge to edge. The bands were symmetrical from outer edge to outer edge.

2) The darker bands show less birefringence.

3) The birefringence was less in the heat treated sample in all areas except the center, area 6, which had the same birefringence for both samples.

4) Through birefringence measurements we have shown the heat treatment does alter the material in some way.

APPENDIX 5

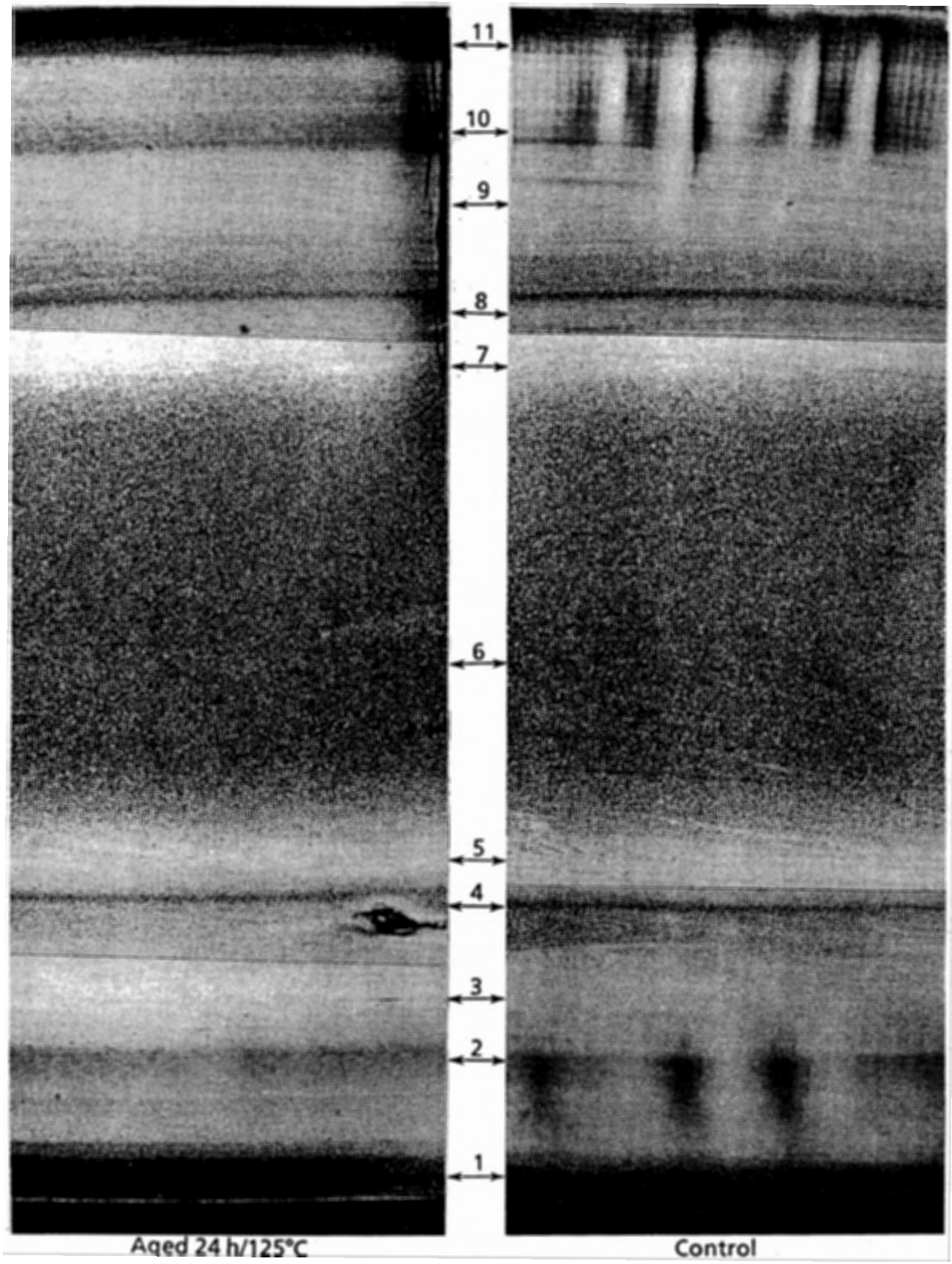
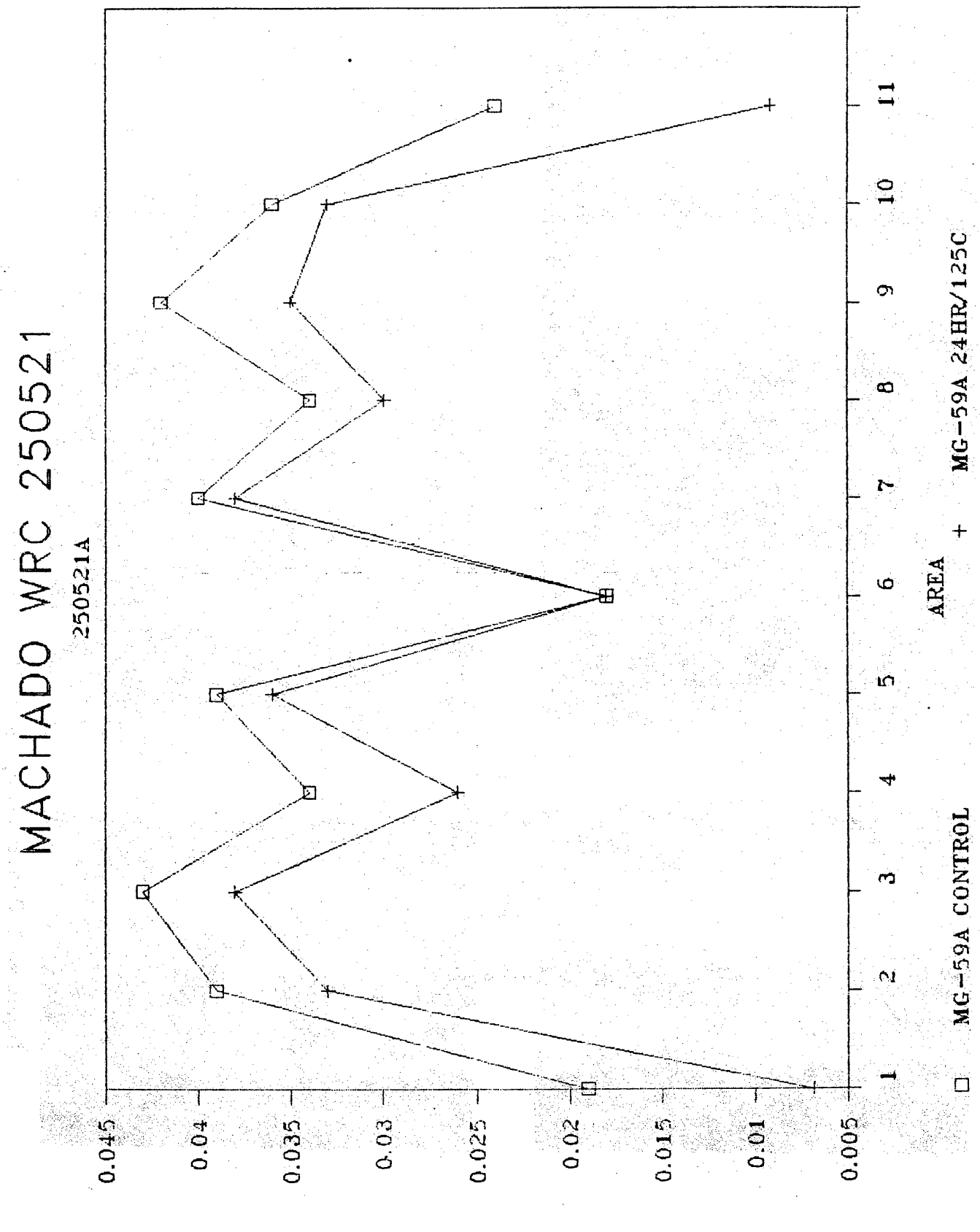


Figure A4-1. Optical Micrographs of Cross-Sections of Unaged and Aged Specimens, Illustrating Areas from which Birefringence Measurements were Taken


MACHADO WRC 250521
250521A
BIREFRINGENCE
0.045
0.04
0.035
0.03
0.025
0.02
0.015
0.01
0.005
1
2
3
4
5
6
7
8
9
10
11
AREA
□ MG-59A CONTROL
+ MG-59A 24HR/125C

APPENDIX 5

OPTICAL SPECTROSCOPY ANALYSIS REPORT

REQUEST NUMBER: WRC250521	SUBMITTER: MACHADO, J.
CHARGE NUMBER: 7326215108	METHOD CODE: 2IRINTA
CONTACTS: RMI	HAZARDS: NONE
DATE REC: 12/15/89	COMPL. DATE: 01/30/90

SAMPLE DESCRIPTION(S):

1) MG59A-Control Carilon
2) MG59A-24hr/125C Carilon

COMMENTS: Analyze bulk and surface portions of each sample. See Courtney Delaney (7792-Microscopy) for samples.

OTHER INSTRUCTIONS: Use IR microprobe to ascertain if chemical degradation is occuring upon oven aging. Look for degradation vs crystallinity effects.

ANALYTICAL RESULT SUMMARY:
Using IR microprobe analysis we compared oven aged and control tensile bars at outermost and centermost locations. We could find no evidence for degradation anywhere in either bar. We find that polyketone crystallinity was much higher in the center of the tensile bars than in the outside skin of the bar in both control and oven aged samples. CARILON crystallinity was modestly increased by the oven aging process in the two locations observed.

RESULTS:
The infrared spectra were examined for any evidence of degradation of the polymer. This can often be observed in the O-H stretching region (near 3400 cm^{-1}) or in the C=O stretching region (near 1700 cm^{-1}). A visual inspection of the data did not reveal evidence for such structures as additional absorptions could not be found in these regions; computer subtraction of surface-bulk for either sample did not turn up any significant differences. Polymer degradation did not appear significant in the IR.

There were significant differences between IR spectra of the skin and bulk CARILON in either sample. In particular, IR bands at 1335, 1053, and 820 cm^{-1} were relatively less intense (around 20% less) in the first 150 microns into the sample than at the center of the sample. These absorptions have previously been shown to be due to crystalline polyketone. They are known to disappear upon conversion to amorphous polyketone, and are accompanied by the growth of IR absorption at 1350, 1070, and 830 cm^{-1} from this phase. This effect was observed in both the control and oven-aged samples. Our conclusion, therefore, is that in the first 150 microns of the sample the polyketone is around 20 percent less crystalline than in the bulk. No other

significant differences between surface and bulk could be seen.

Comparing the oven aged to the control sample in either region yielded a similar difference in IR bands and no other. In comparing IR spectra of either the first 150 microns or the center, we observe that bands from crystalline polyketone are higher in the oven aged material. We conclude, thus, that crystallinity is enhanced by the low temperature oven aging.

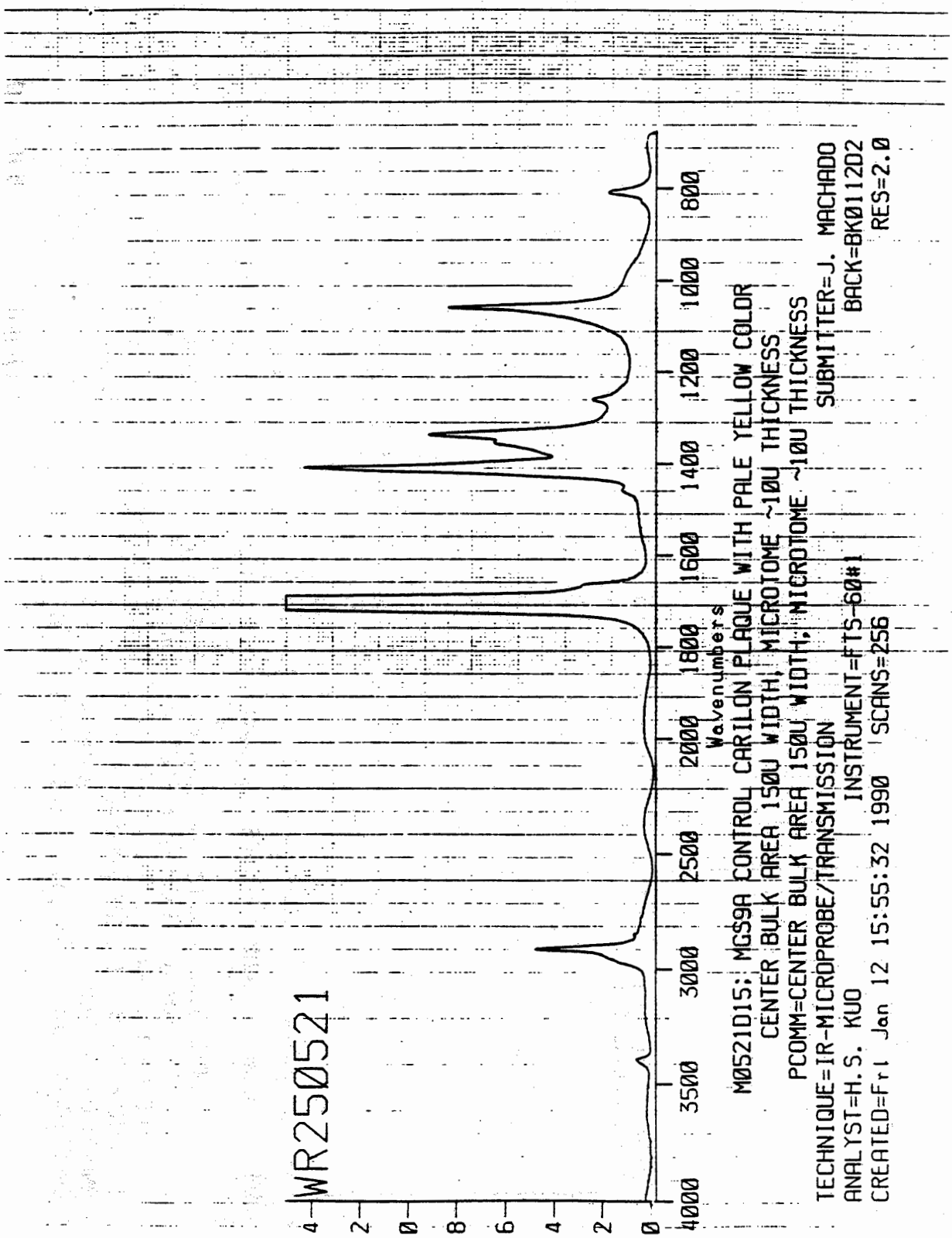
No other significant differences could be discerned.

EXPERIMENTAL:
The Digilab UMA300 IR microprobe was utilized for the analysis. Thin cross sections, around 10 microns in thickness, were cut by the microscopy lab for our analysis. We analyzed retangular polymer regions, 150x250 microns in size, at the outside edge and center of the samples. Our outside spectra, thus, are representative of the polymer from 0 to 150 microns in depth from the surface.

If any additional information is needed, please contact me.

Rich Irwin (7089, PROFS=RMI)

[WRC250521; MACHADO, J.; 7326215108; RMI; HSK; FTS60#1; M0521D1*, M0521D2*; 2IRINTA; 01/30/90 ; CARILON, DEGRADATION, OVEN AGING, CRYSTALLINITY CHANGE]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Wavenumbers
M0521D15; MG59A CONTROL CARILON PLAQUE WITH PALE YELLOW COLOR
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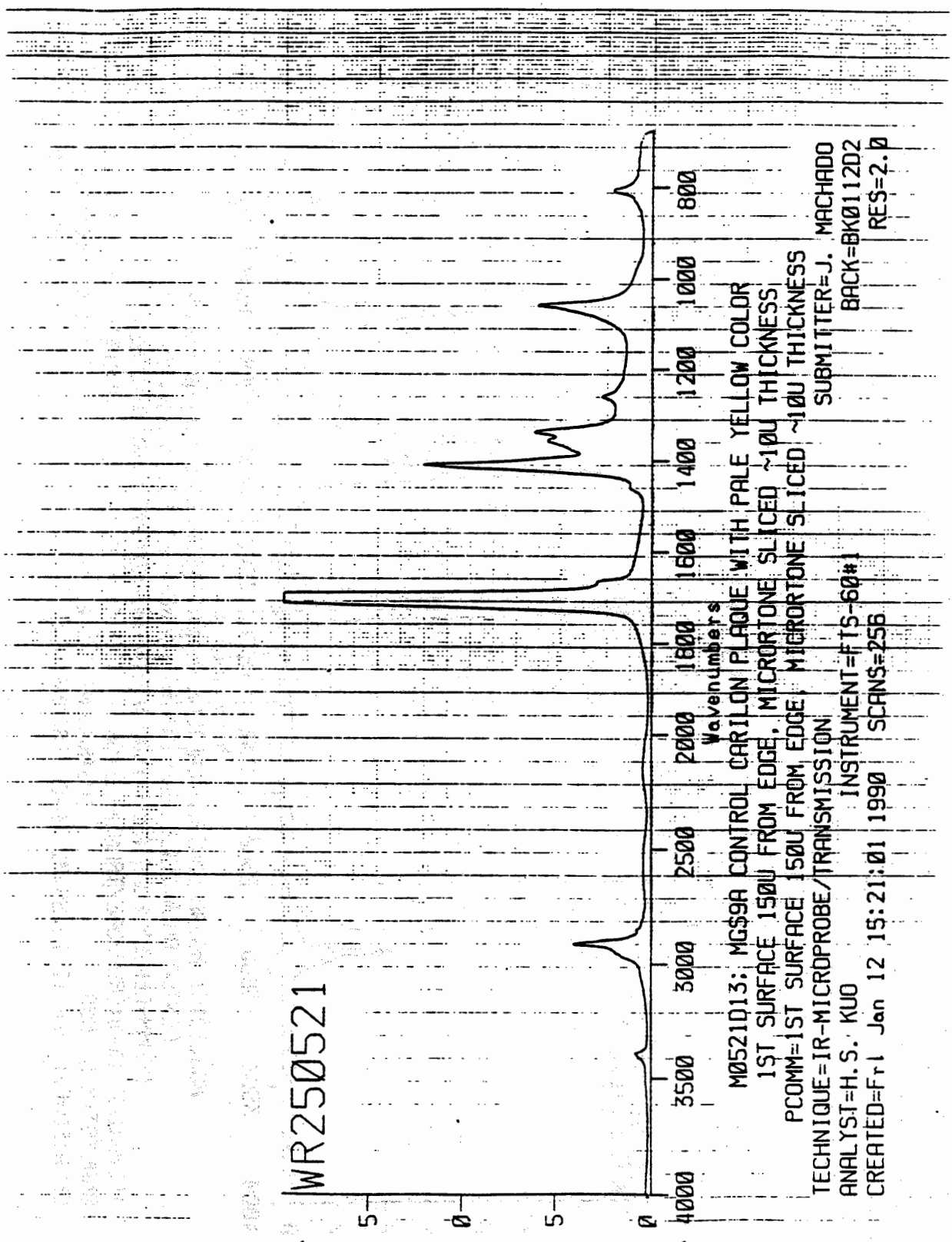
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PCOMM=1ST SURFACE 150U FROM EDGE, MICRORTONE SLICED ~10U THICKNESS
TECHNIQUE=IR-MICROPROBE/TRANSMISSION SUBMITTER=J. MACHADO
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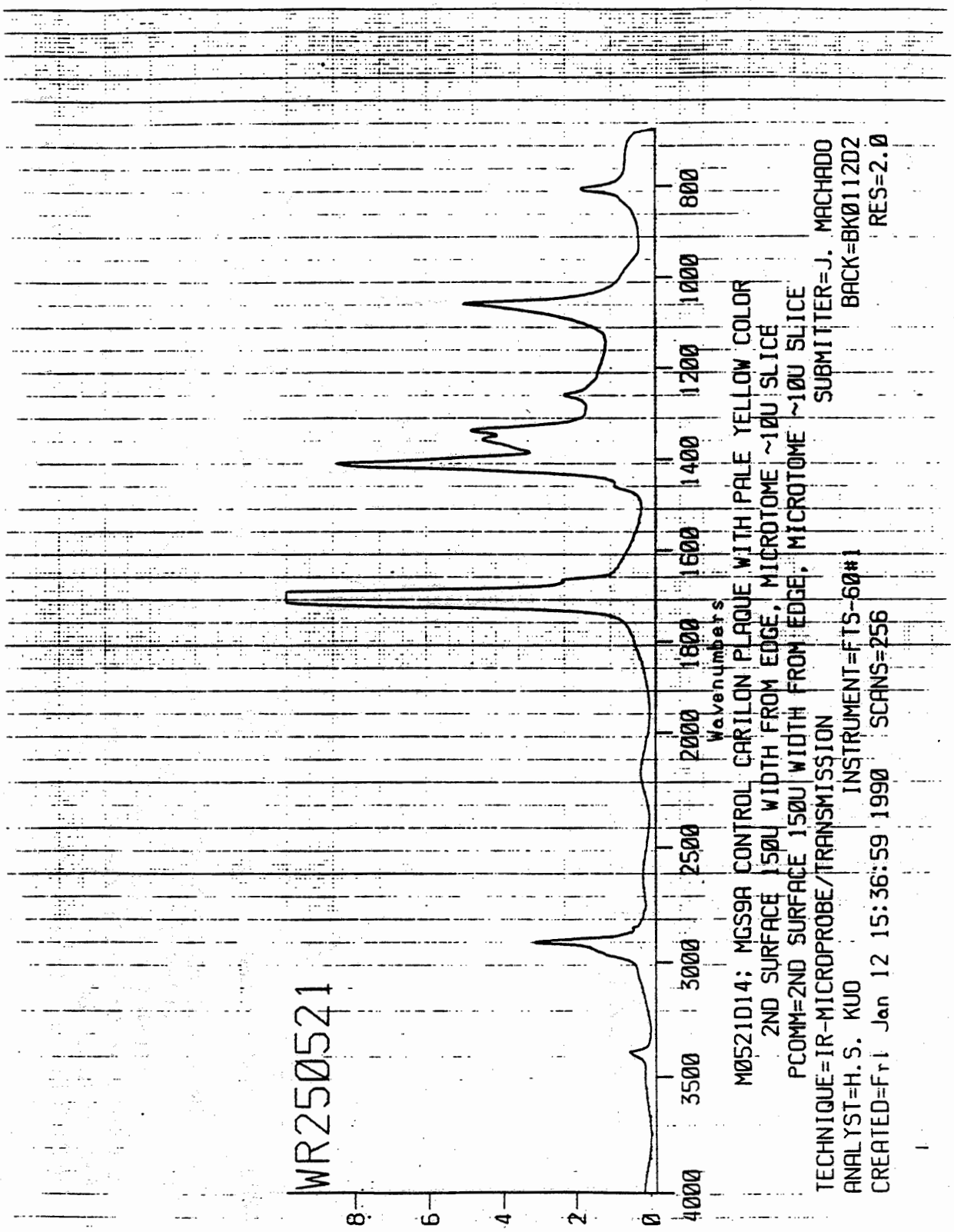
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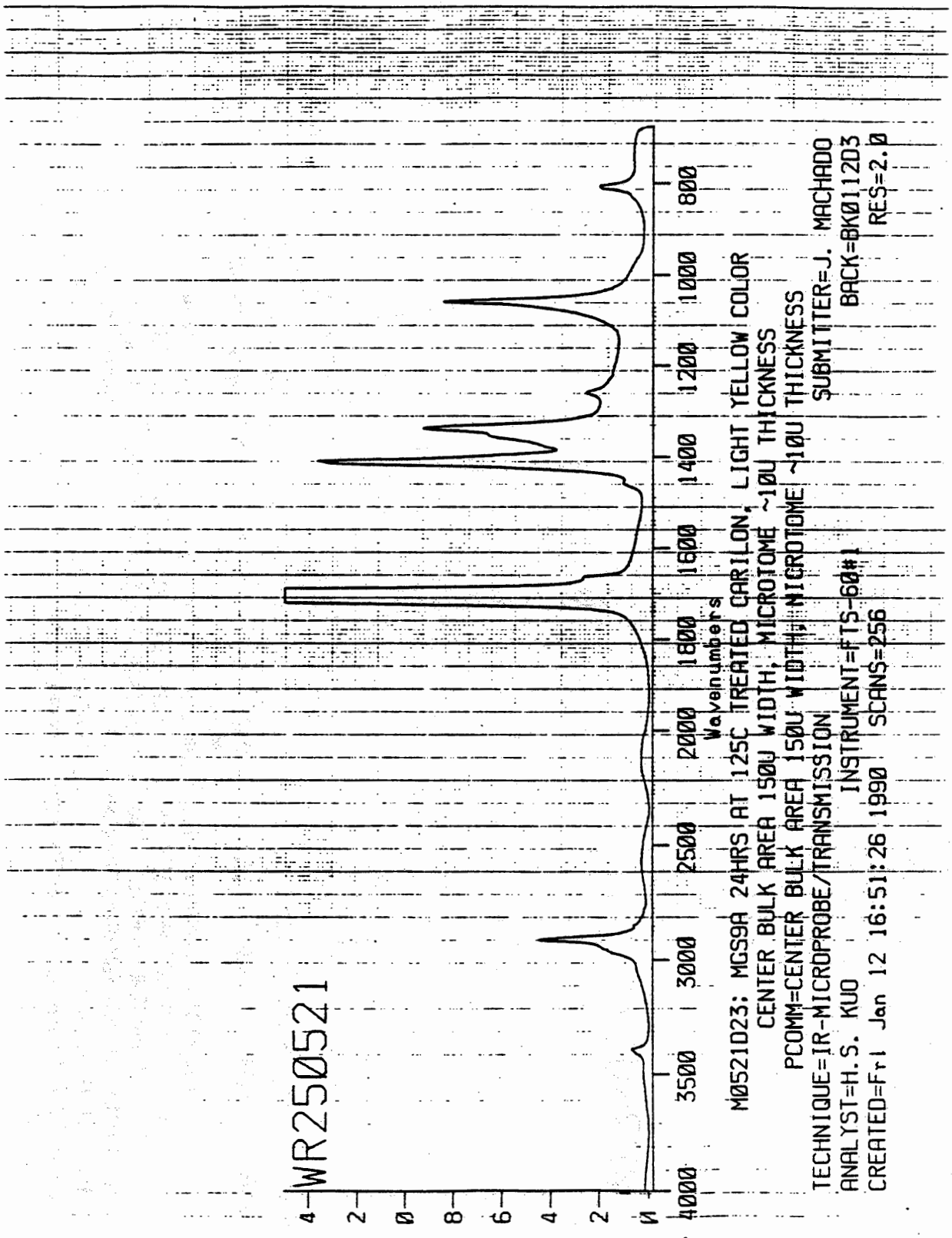
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Wavenumbers
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PCOMM=CENTER BULK AREA 150U WIDTH, MICROTOME ~10U THICKNESS
TECHNIQUE=IR-MICROPROBE/TRANSMISSION SUBMITTER=J. MACHADO
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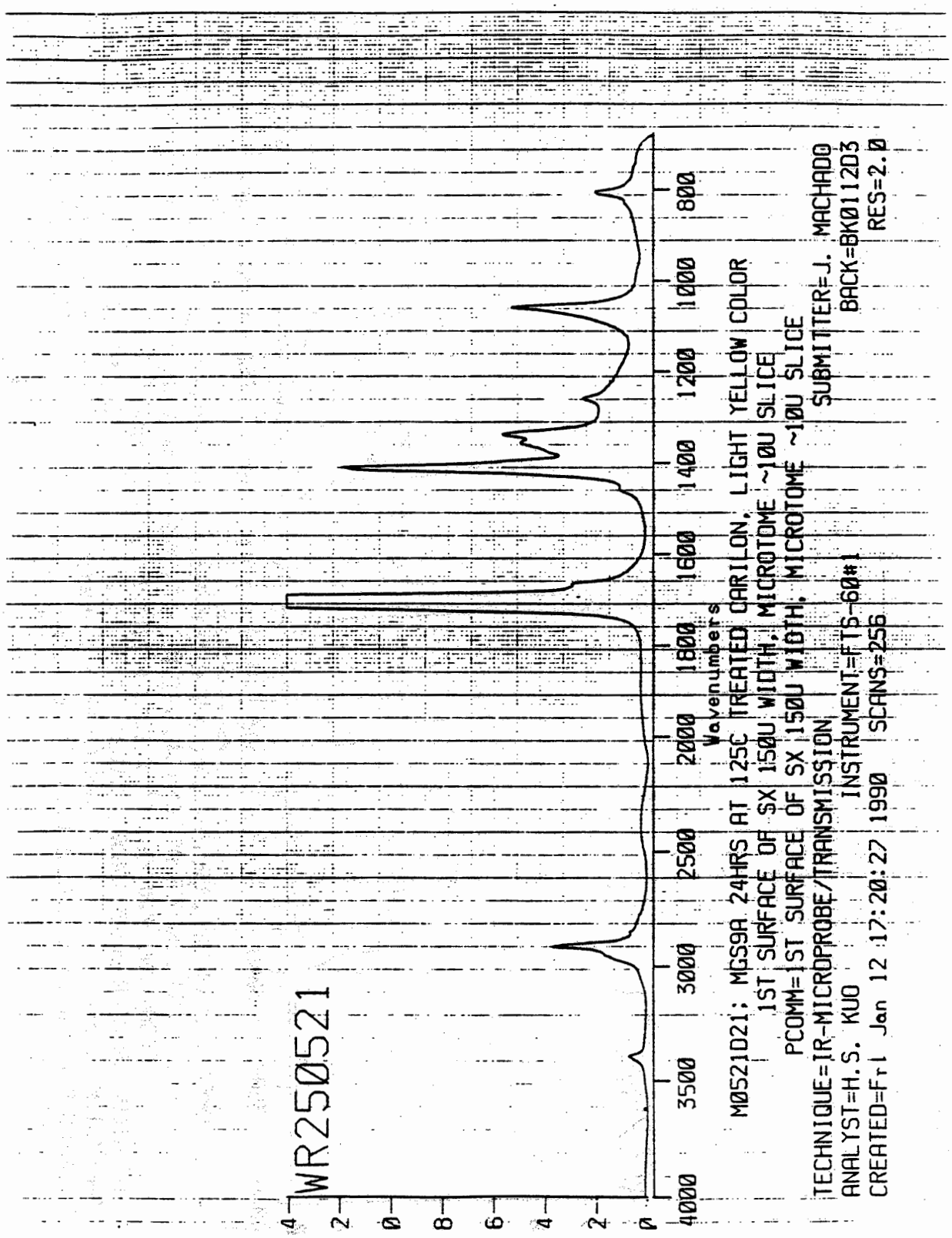
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PCOMM=1ST SURFACE OF SX 150U WIDTH, MICROTOME ~10U SLICE
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ANALYST=H.S. KUO
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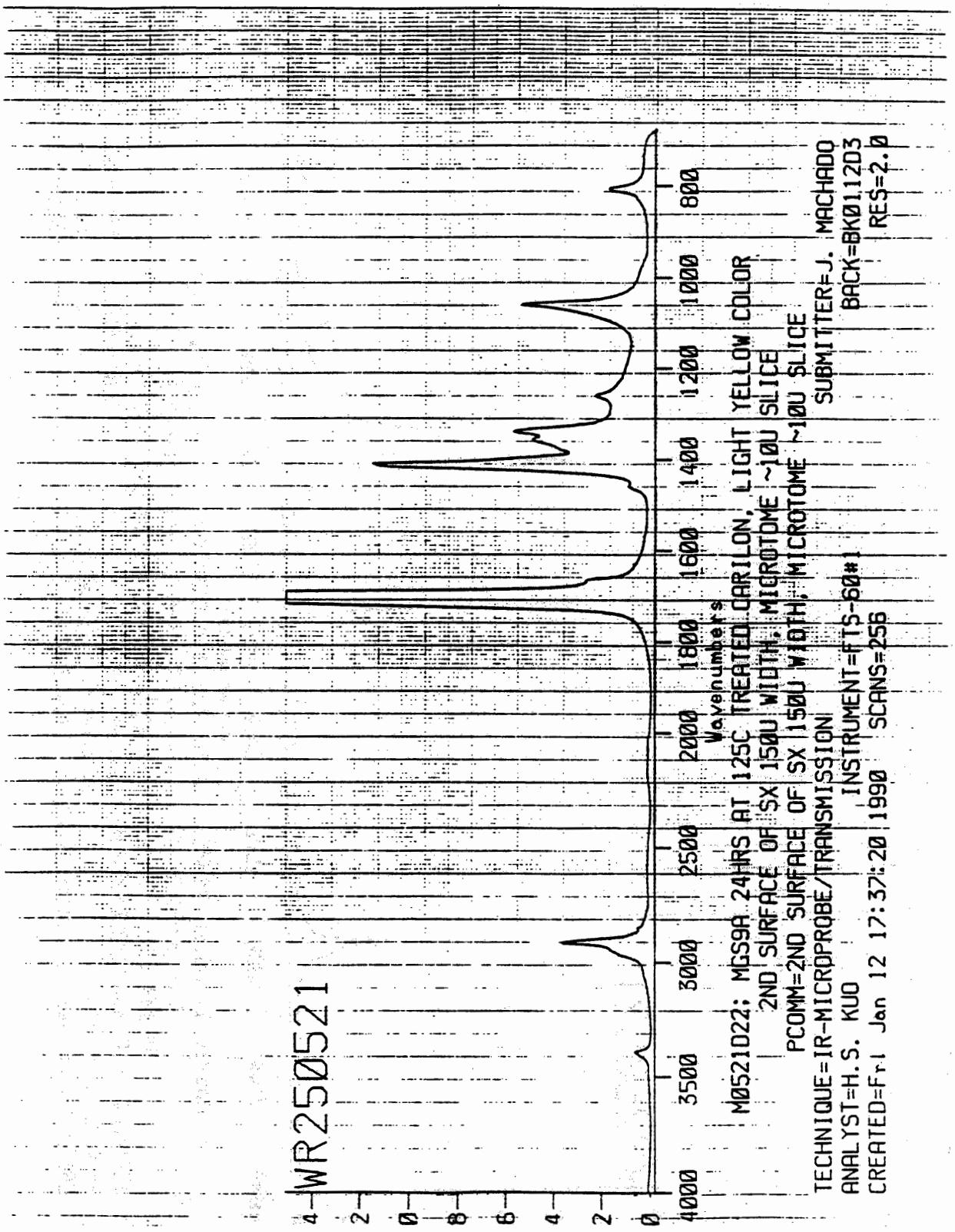
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Wavenumbers
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M0521D22; MS9A 24HRS AT 125C TREATED CARILON, LIGHT YELLOW COLOR
2ND SURFACE OF SX 150U WIDTH, MICROTOME ~10U SLICE
PCOMM=2ND SURFACE OF SX 150U WIDTH, MICROTOME ~10U SLICE
TECHNIQUE=IR-MICROPROBE/TRANSMISSION
ANALYST=H.S. KUO
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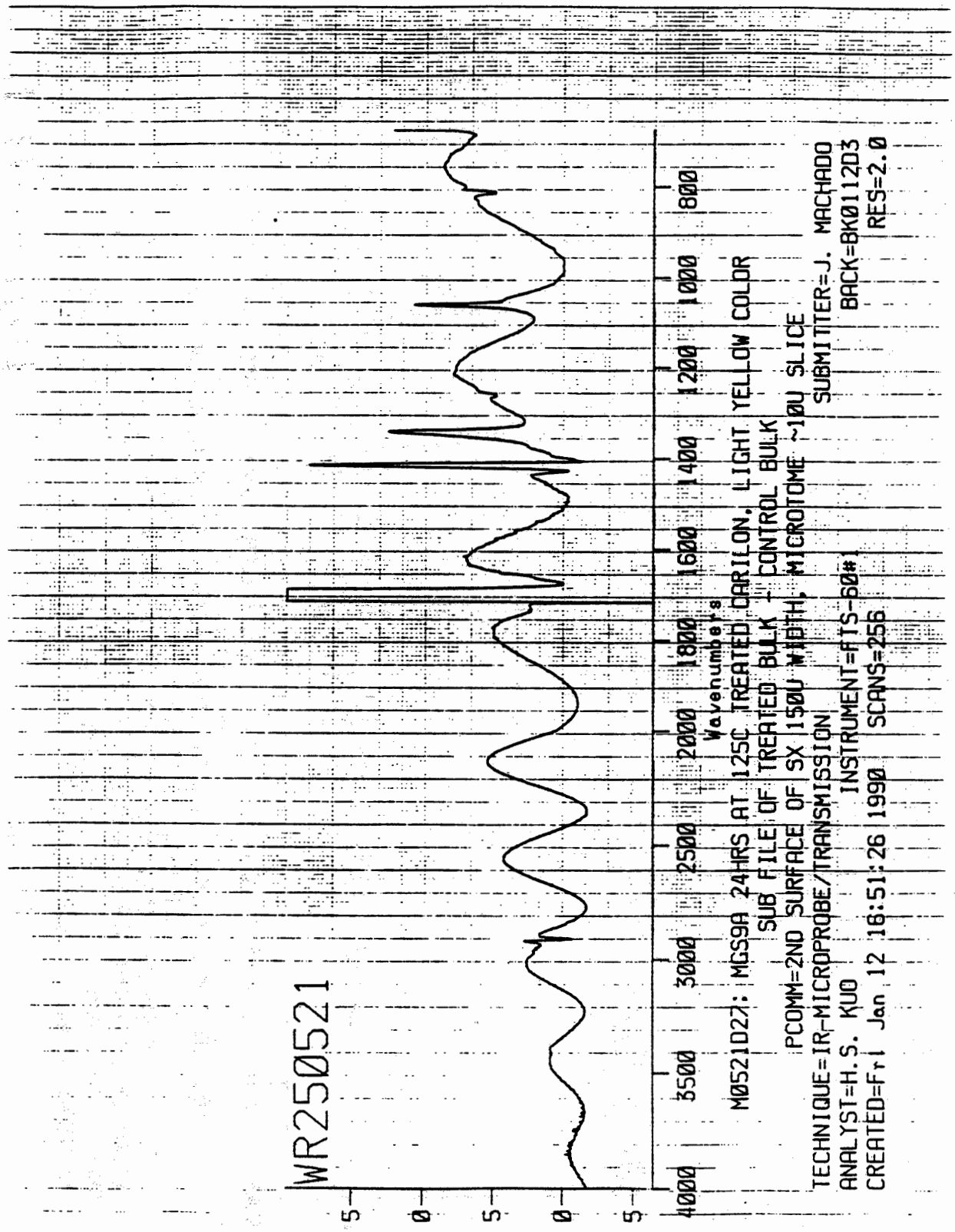
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Wavenumbers
M0521D27: MGS9A 24HRS AT 125C TREATED CARILON, LIGHT YELLOW COLOR
SUB FILE OF TREATED BULK - CONTROL BULK
PCOMM=2ND SURFACE OF SX 150U WIDTH, MICROTOME ~100U SLICE
TECHNIQUE=IR-MICROPROBE/TRANSMISSION
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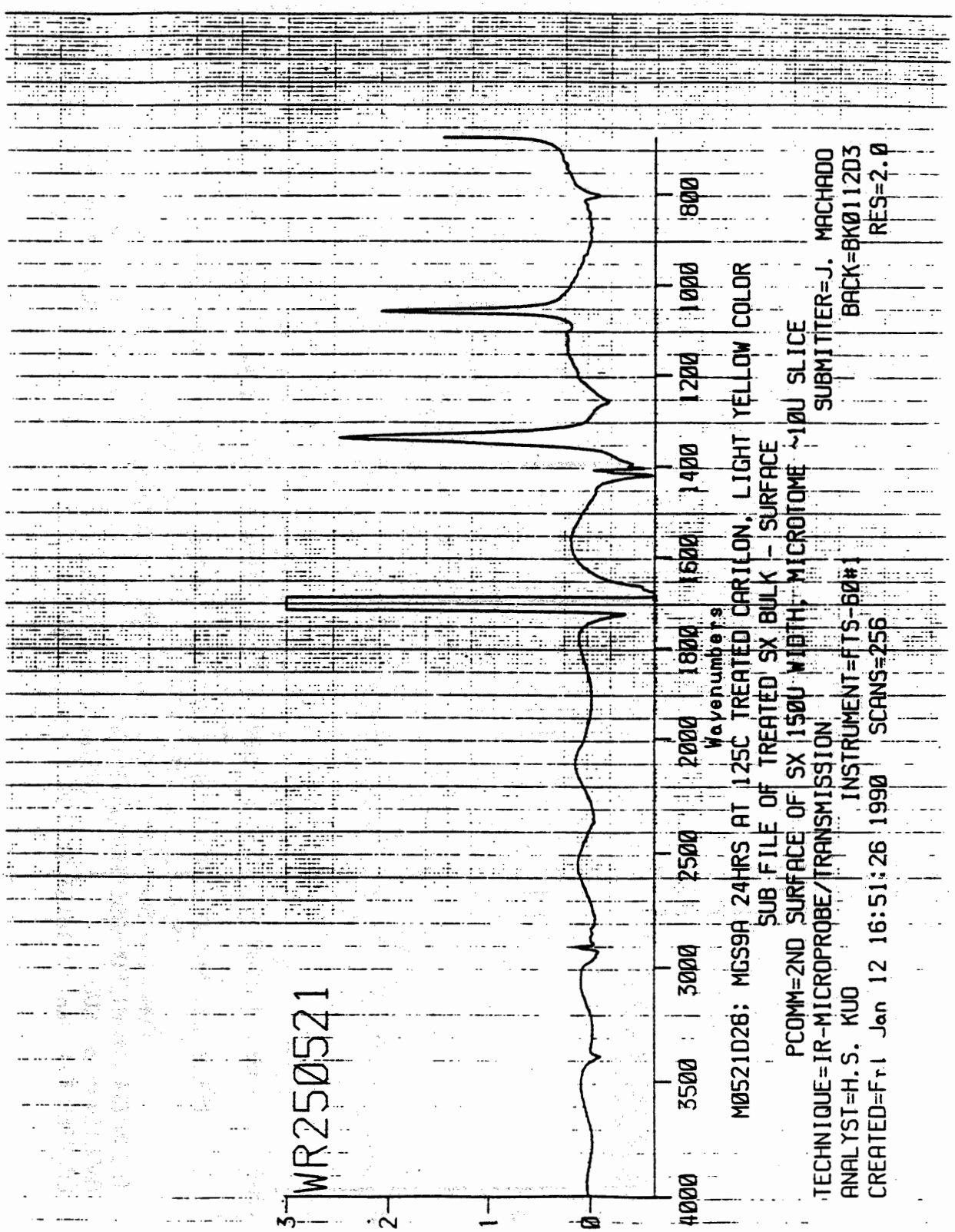
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SUB FILE OF TREATED SX BULK - SURFACE
PCOMM=2ND SURFACE OF SX 150U WIDTH, MICROTOME ~10U SLICE
SUBMITTER=J. MACHADO
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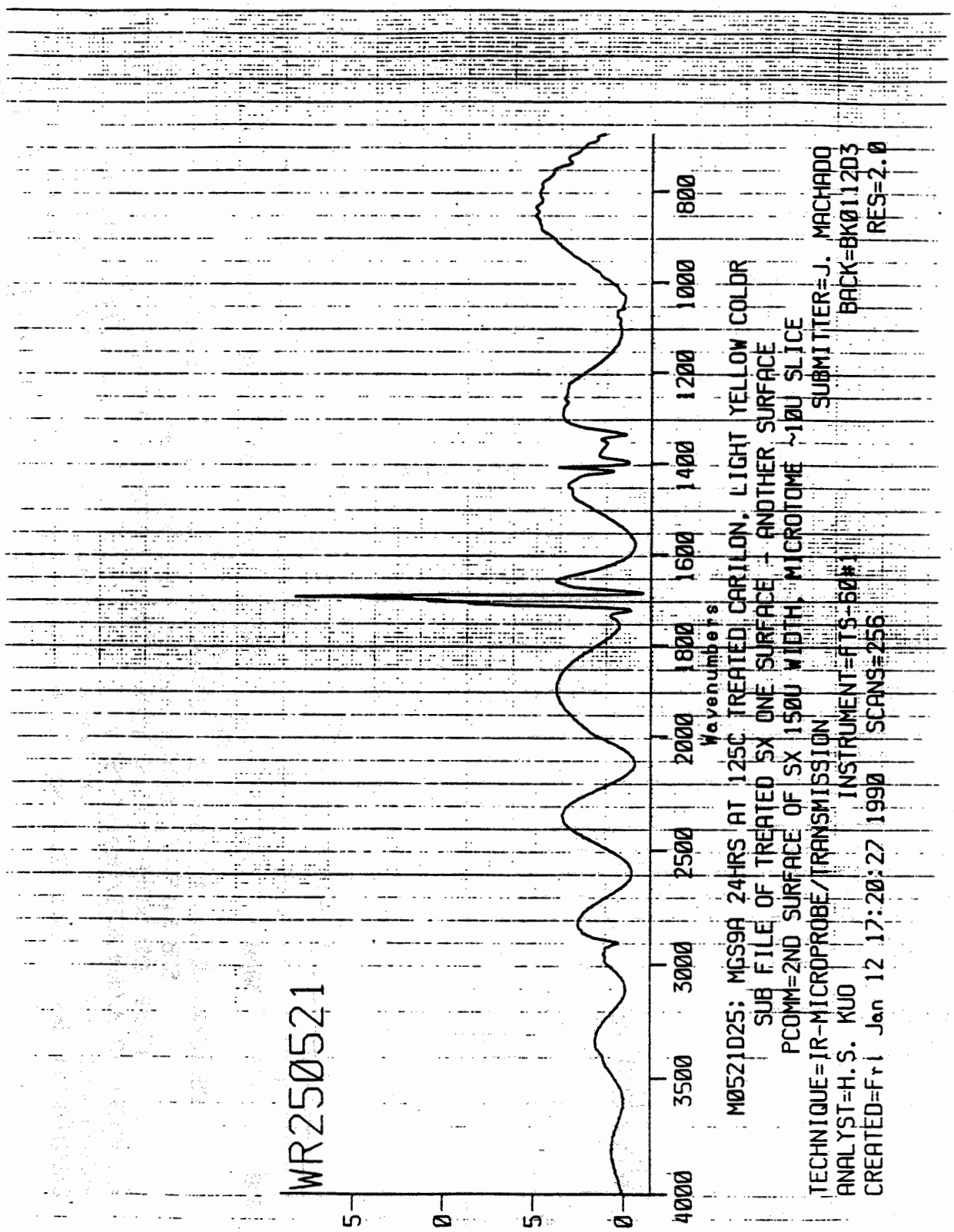
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Wavenumber's
M0521D25; MGS9A 24HRS AT 125C TREATED CARILON, LIGHT YELLOW COLOR
SUB FILE OF TREATED SX ONE SURFACE + ANOTHER SURFACE
PCOMM=2ND SURFACE OF SX 150U WIDTH, MICROTOME ~10U SLICE
TECHNIQUE=IR-MICROPROBE/TRANSMISSION SUBMITTER=J. MACHADO
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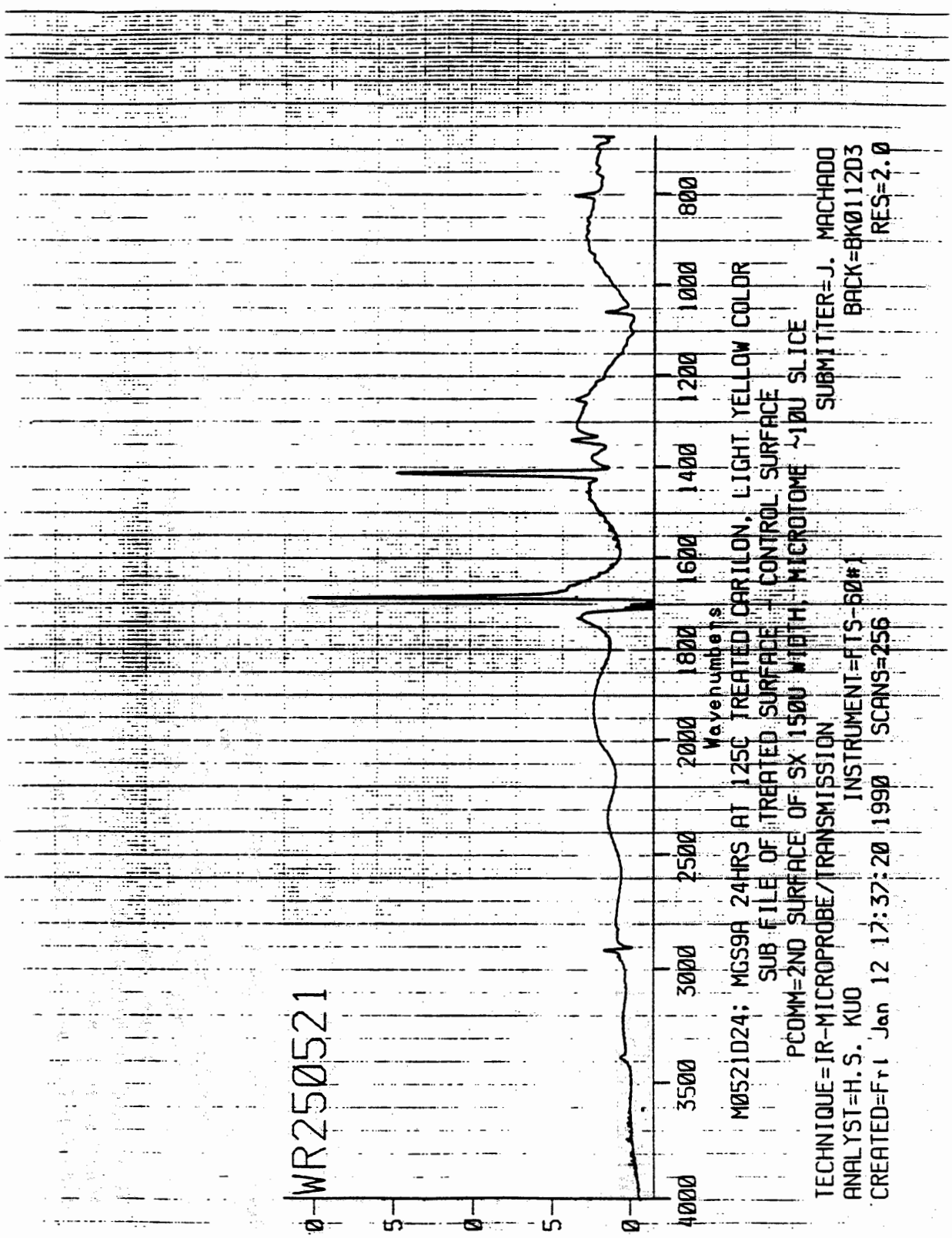
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Wavenumbers
M052104: MGS9A 24HRS AT 125C TREATED CARILON, LIGHT YELLOW COLOR
SUB FILE OF TREATED SURFACE - CONTROL SURFACE
PCOMM=2ND SURFACE OF SX 150U WIDTH, MICROTOME ~10U SLICE
TECHNIQUE=IR-MICROPROBE/TRANSMISSION
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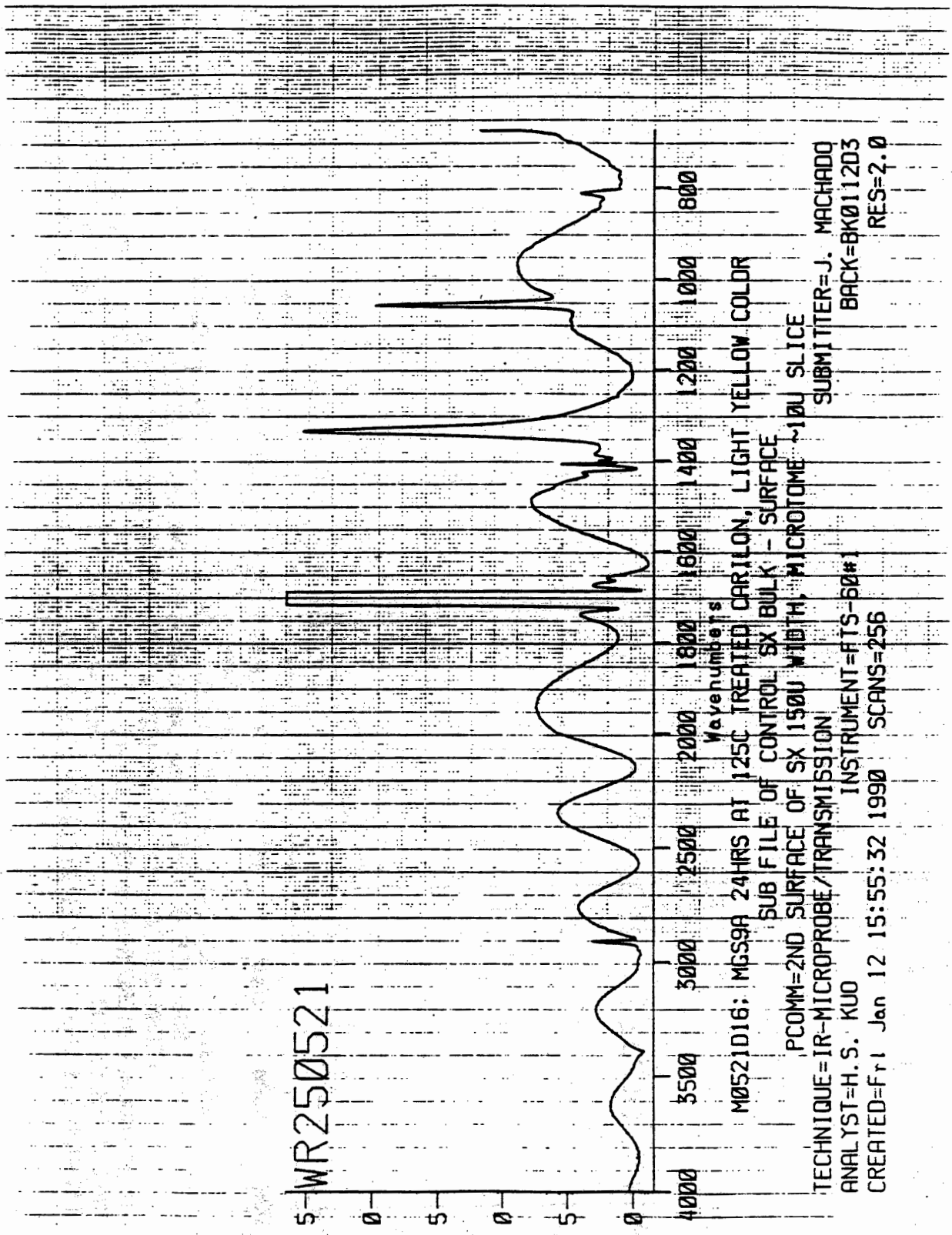
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Wavenumbers
M0521D16: MGS9A 24HRS AT 125C TREATED CARILON, LIGHT YELLOW COLOR
SUB FILE OF CONTROL SX BULK - SURFACE ~10U SLICE
PCOMM=2ND SURFACE OF SX 150U WIDTH, MICROTOME ~10U SLICE
TECHNIQUE=IR-MICROPROBE/TRANSMISSION
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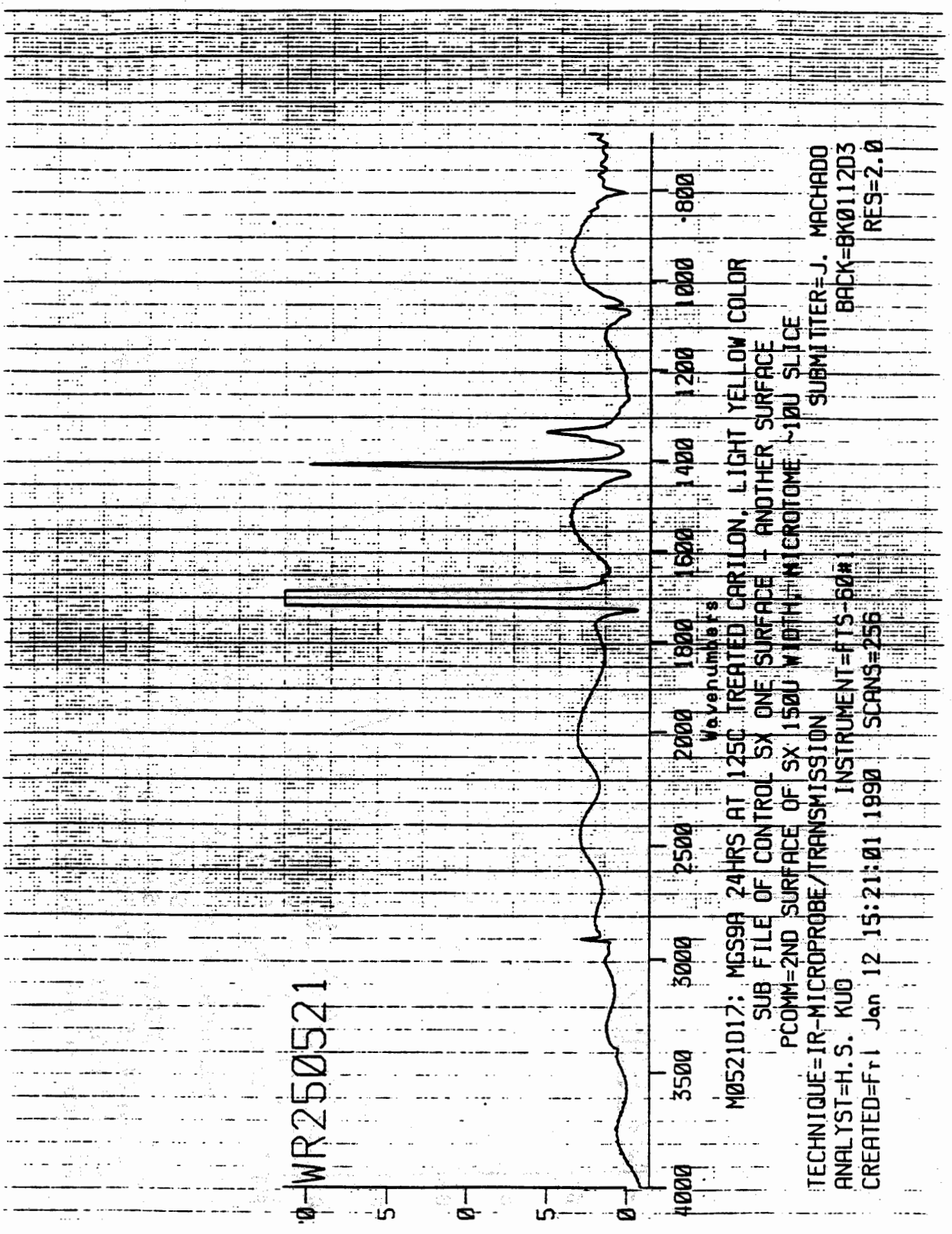
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Wavenumbers
M0521D17; MGS9A 24HRS AT 125C TREATED CARILON, LIGHT YELLOW COLOR
SUB FILE OF CONTROL SX ONE SURFACE + ANOTHER SURFACE
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