

**COAL PRECURSORS FOR PRODUCTION OF  
CARBON AND GRAPHITE PRODUCTS**

**FINAL REPORT**

April 8, 1996

Report Prepared by  
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under Subcontract 62X-SR928C, WBS Element-UCAR-3

for  
OAK RIDGE NATIONAL LABORATORY  
Oak Ridge, Tennessee 37831  
Managed by  
LOCKHEED MARTIN ENERGY RESEARCH CORPORATION  
for the  
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Research Sponsored by the U.S. Department of Energy,  
Office of Fossil Energy  
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## I. SUMMARY

Chemical and analytical characterization data were obtained for NMP extracts of hydrotreated coal prepared by the West Virginia University (WVU) Group. Initial evaluation of small-scale samples produced by varying coal hydrotreating time, temperature, and catalyst type were used to define the conditions that would produce material suitable as binder, impregnant, C/C matrix pitches and precursors for needle coke, anode coke, and mesophase pitch fibers. In addition to analytical characterization, mesophase formation studies were used to assess the coal extract quality.

Based on the small-scale testing, specific extracts derived from hydrotreating coal at 450°C were recommended for scaleup. Evaluation of the 450°C - 2-hour hydrotreated coal extract showed that it should be suitable as an impregnant or binder pitch and as a precursor for needle coke and mesophase pitch. This material formed a large-domained mesophase, despite a high oxygen and nitrogen content.

A small graphite artifact was produced using coke derived from the extract as the filler and the extract itself as the binder. The coefficient of thermal expansion (CTE) of  $0.26 \times 10^{-6}/\text{C}^\circ$ , measured for the artifact is in the range found for electrode type graphite.

Coal extraction residues were steam activated to produce active carbons with surface areas from 400-700 m<sup>2</sup>/gm. The program has demonstrated that the coal extraction process coupled with hydrotreatment, has the potential for providing a broad range of carbon and graphite products.

## II. INTRODUCTION

The main goal of the overall program on "Coal-Based Precursors for Carbon Products" was to demonstrate the utility of coal extracts from the WVU extraction process as suitable base raw materials for the carbon products encompassed by the Carbon Products Consortium (CPC) team. These include binder and impregnating pitch, coke for graphite electrodes, cokes for anodes and specialty graphite, matrices for C/C composites and raw material for mesophase pitch fibers. Each of these products has unique requirements for properties of a suitable precursor. Our previous work on the Coal Precursor for Graphite Program<sup>(1)</sup> has shown that the WVU coal extraction process coupled with hydrotreatment, does have the potential for achieving this objective. Our current effort, therefore, involved the screening and evaluation of extracts produced by the WVU Group and recommending appropriate materials for scaleup for subsequent evaluation by Consortium Team members.

Our program involved an initial characterization of small-scale extracts using standard analytical methods and mesophase formation studies. This was followed by feedback to the WVU Group and to the CPC partners (Koppers, ALCOA, FMI, and APPI) with recommendation of material for scaleup. We also performed similar analytical and mesophase studies on some of the scaled-up extracts.

<sup>(1)</sup>Research sponsored by the U.S. Department of Energy, Fossil Energy Advanced Research and Technology Development Materials Program, DOE/FE AA 15 10 10 0, Work Breakdown Structure Element UCAR-3"

As part of our program, we investigated the activation of the coal extraction residues for the purpose of producing a useful active carbon. A further task, which was started towards the end of the program, was to fabricate a small graphite artifact using coke derived from coal extract as the filler and the coal extract itself as a binder. The results of our studies are summarized in the following sections of this report.

### **III. EXPERIMENTAL PROCEDURES**

#### **A. Analytical Methods**

1. Elemental Analysis - Elemental C, H, N, and O data were measured using a LECO CHN 1000 Analyzer. Elemental sulfur was measured using a LECO SC-132 carbon analyzer.
2. Thermogravimetric Analysis (TGA) - The data were obtained using TA Instruments 951 TGA module. Samples were heated at a rate of 10°C/minute in an inert atmosphere.
3. Nuclear Magnetic Resonance (NMR) - The NMR spectra were measured for CS<sub>2</sub> solutions using a Varian A-60 spectrometer. In order to increase solubility, the solutions were prepared by either refluxing with solvent or by ultrasonic mixing.
4. Toluene and Trichlorobenzene (TCB) Solubility - The insoluble contents in toluene and TCB were determined using procedures in ASTM D-4072.
5. Mettler Softening Point (SP) - The softening points were measured using a Mettler FP83HT SP apparatus following the standard procedures in ASTM D-3104.
6. Modified Conradson Carbon (MCC) - The MCC data (carbon yields) were obtained using procedures in ASTM D-2146.
7. Gel Permeation Chromatography (GPC) - Molecular weight determinations were obtained from GPC measurements using a Dupont 850 Liquid Chromatographic System coupled with a Knauer High Temperature Differential Refractometer. The column configuration and calibration have been described elsewhere<sup>(2)</sup>. The GPC data were obtained for the portion soluble in TCB solvent at 90°C.

## B. Mesophase Formation Studies

### 1. Test-Tube Evaluations

The small-scale extracts were evaluated initially for mesophase quality by heating 0.7-1.3 gm samples in pyrex test tubes for six hours at 400°C in an inert gas environment. The objective was to produce a 15-40% mesophase content in the heat-treated residue, with most of the mesophase concentrated at the bottom of the test tube. After heat treatment, the pyrex test tubes were dissolved in aqueous HF, and the residues were mounted and polished by standard metallographic procedures to show a midline vertical cross-section. Polarized light microscopy techniques were used to make a qualitative assessment of the amount and quality of the mesophase and the presence, if any, of insoluble solid contaminants in the extracts.

### 2. Mesophase Domain Size

Specimens for automated image analysis were prepared by heat treating the coal extracts in an inert atmosphere at 400°C in ceramic boats and then preparing "annealed" samples for the mesophase domain size measurements using procedures described elsewhere<sup>(3)</sup>.

### 3. Hot-Stage Microscopy

Direct observations of the melting behavior, gas evolution characteristics, mesophase formation rate, and mesophase quality were made while heating the coal extracts up to 400°C at 6-10°C/min. and holding at 400°C for several hours. Details of the cover glass "sandwich" configuration used to contain the sample, the modified Kofler hot stage and the polarized light microscope have been reported previously<sup>(4)</sup>.

A simple CCD video camera and color monitor/VCR system were acquired late in the contract period to facilitate direct recording of the dynamic behavior of the coal extracts when they were heated on the hot-stage microscope.

## C. Steam Activations

Activation experiments were carried out on powdered coal extraction residues using a specially designed tumbling furnace. The activations were performed by passing a mixture of steam and N<sub>2</sub> over the sample maintained at a constant temperature of 900°C. The treatment was interrupted intermittently and the sample weighed to determine percent burn-off.

#### D. Graphite Fabrication

Small 19mm-diameter graphite electrodes were fabricated using calcined coke produced from hydrotreated coal extract as filler and the hydrotreated extract as the binder. The binder and filler were mixed and extruded using standard procedures. The formed rods were then baked and graphitized to 3,000°C.

### IV. EXPERIMENTAL RESULTS

#### A. Small-Scale Coal Extracts

##### 1. Sample Preparations

The WVU Group initially carried out a series of small-scale extractions on a hydrotreated bituminous coal, WVGS-13421. The coal was hydrotreated at three different temperatures 350°C, 400°C, and 450°C for a period of 1 hour in 800 psig H<sub>2</sub>. An additional set of hydrotreatments was carried out at 450°C for 2 hours. For each temperature, the hydrotreatments were performed using tetralin as a hydrogen donor without a catalyst, and with the use of a Fe<sub>2</sub>S<sub>3</sub> catalyst or a sulfided Mo catalyst. The NMP extractions were performed on the hydrotreated products.

Initial characterization data from the WVU Group showed that the softening temperatures of the 350°C and 400°C extracts were much too high to make them useful for most of the proposed applications of the Team. We therefore agreed to fully characterize only the 450°C-treated materials.

Our characterization results for the extracts from the WVGS-13421 coal hydrotreated at 450°C for 1 and 2 hours follow.

##### 2. Analytical Characterization of 450°C - 1-Hour Samples

The analytical characterization data for the 3 different materials prepared from hydrotreated coal at 450°C for 1 hour are presented in Table I. The Mettler softening points (measured at WVU) range from 156° to 190°C which are too high for use as conventional binder and impregnant pitches.



**Table I**  
**Characterization Data for Extracts from Hydrotreated Coal, WVGS-13421, 450°C - 1 Hour**

	C-269-2 Thermal	C-279-1 Fe <sub>2</sub> S <sub>3</sub>	C-288-2 Mo(S)
SP°C	190	156	158
%C	85.2	84.5	81.9
%H	5.51	5.63	5.82
C/H atomic ratio	1.30	1.26	1.18
%N	3.08	2.89	3.73
%O	4.73	4.60	6.07
%S	0.49	0.57	0.43
TGA Yd%	49	46	42
%TI	28.9	-----	-----
%Arom. H.	38	36	33
GPC - MW (Mn)	415	395	371
%TCB Insoluble	40.6	26.0	19.7
%NMP	0	0.1	5

All the extracts show very high N and O contents. The Mo(S)-treated coal extract showed the highest N content as a result of retained NMP. The low C/H ratios are expected for the hydrotreated extracts.

The proton NMR spectra obtained in CS<sub>2</sub> solution are presented in Figures 1-3. The extracts were incompletely soluble in CS<sub>2</sub>. We were able to obtain essentially complete solution in pyridine D-5; however, the residual hydrogen or water in the solvent interfered with the analysis. The extracts all show extremely high aliphatic hydrogen contents resulting from the hydrogenation of aromatic rings. The NMR results are also summarized in Table I. For comparison, a spectrum for coal tar binder pitch exhibits about 85% aromatic hydrogen compared to the 33-38% range for the extracts. From the NMR data as well as the C/H ratios, it appears that the catalytically treated materials may have been slightly more hydrogenated.

The TGA data for the 3 extracts are presented in Figures 4-6. The TGA carbon yields of 42-49% are quite acceptable for a pitch-type material. The low-temperature weight loss occurring from about 150-250°C is attributed to evolution of retained NMP solvent. This is particularly apparent in the TGA result for the Mo(S) product in Figure 6.

The GPC curves for the three 450°C - 1-hr. samples are shown in Figures 7-9. They are all similar, except for a resolved doublet in the Fe<sub>2</sub>S<sub>3</sub>

catalyzed material. They all show a broad symmetrical molecular weight distribution (MWD) with an apparent molecular weight range of about 250-900. The negative peaks in the GPC spectra are attributed to retained NMP. An estimate of the amount of NMP present was made by integrating these peaks and the results are summarized in the last line of Table I. Also shown in Table I are the toluene insolubles (TI) content and the measured insolubles in TCB.

3. Analytical Characterization of 450°C - 2-Hour Samples

The analytical data for the small-scale extracts obtained from coal hydrotreated at 450°C for 2 hours are summarized in Table II. The softening points are all considerably lower than those for the samples produced by hydrotreating for 450°C for 1 hour. Like the 450°C - 1-hour samples, the products in Table II exhibit very high N and O contents. The H contents, however, are higher, for the 2-hour samples reflecting the greater degree of hydrogenation. The 2-hour materials also showed a higher solubility in TCB than the 1-hour samples.

**Table II**  
**Characterization Data for Extracts from Hydrotreated Coal,**  
**WVGS-13421, 450°C - 2 Hours**

	C-300-3 Thermal	C-300-6 Fe <sub>2</sub> S <sub>3</sub>	C-300-9 Mo(S)
SP°C	126	123	131
%C	84.2	83.9	86.1
%H	5.74	5.75	5.82
C/H atomic ratio	1.23	1.23	1.27
%N	3.65	3.60	3.22
%O	5.53	5.66	4.61
%S	0.35	0.42	0.41
TGA Yd%	40	41	42
%Arom. H	40	40	43
GPC - Mn	421	426	416
%TCB Insoluble	21.9	22.3	18.1
%NMP	2	2	1

The proton NMR data for the extracts in Figures 10-12 show a lower content of long chain aliphatic protons than for the 1-hour treated samples, indicative of more selective hydrogenation of aromatic rings. The TGA results in Figures 13-15 demonstrate a reduced carbon yield for the

450°C - 2-hour materials as compared to the 1-hour samples. The presence of retained NMP is evident from the weight loss occurring below 300°C. The GPC curves for the 450°C - 2-hour samples in Figures 16-18 exhibit somewhat broader MWD's when compared to the 1-hour extracts, which could account for the lower softening points. The amounts of NMP solvent present in the extracts, as measured from integrating the negative GPC peaks, are also summarized in Table II. From the measured properties, there appears to be no advantage for the use of a catalyst during the hydrotreatment.

#### 4. Mesophase Formation Studies of 450°C - 1-Hour and 2-Hour Samples

Hot-stage microscopy examinations were carried out on all three of the small-scale 450°C - 1-hour samples. The primary results from the heat-up and hold period at 400°C are summarized in Table III. The initial softening temperatures from visual observations on the hot-stage were reasonably consistent with the Mettler S. P. results in Table I. The most unusual feature of the hot-stage microscopy observations was the onset of vigorous bubbling, presumably gas evolution, at temperatures in the 250° to 275°C range. Conventional coal tar and petroleum pitches do not show this vigorous bubbling behavior below 300°C. Evolution of residual NMP (202°C Boiling Point) could be responsible for a portion of the low-temperature gas evolution from the extracts, but certainly not all of it, because the vigorous gas evolution continued throughout the 300°-400°C range. Dehydrogenation and/or decomposition reactions are suspected to be the origin of the gas evolution in these initial 450°C - 1-hour extracts, but an attempt to confirm evolution of H<sub>2</sub>, H<sub>2</sub>O, or other low MW gases in this temperature range was not successful<sup>(5)</sup>.

Mesophase spheres were detected at the edge of the cover glass sandwich in all three 450°C - 1-hour samples after ~20 min. at 400°C, indicating that all of them had comparable mesophase formation rates. The mesophase viscosity appeared to be reasonably low, based on bubble deformation behavior, and was essentially equivalent in the 3 samples. The overall mesophase quality was estimated to be borderline for a needle coke precursor.

All of the samples contained a small amount of finely divided solids, but none had a concentration judged high enough to have a significant effect on coke quality or performance as an impregnant. However, these finely divided solids would be expected to degrade the performance of these extracts as a mesophase pitch fiber precursor.

All of the 450°C - 1-hour and 450°C - 2-hour samples were evaluated by the test-tube method. Measured yields after heat treatment at 400°C for six hours were in the 72 to 85% range. However, all of these samples foamed extensively during the heat treatment, so some material may have been lost. The extensive foaming during the test-tube heat treatments of the 450°C - 1-hour samples is consistent with the vigorous low-temperature bubbling behavior noted during the hot-stage microscopy observations. The 450°C - 2-hour samples foamed as much as the 450°C - 1-hour samples in the test-tube heat treatments, so the additional reaction time at 450°C did not seem to have any significant effect on this characteristic of the small-scale extracts.

Examination of the domain size in the coalesced mesophase layer, which collects at the bottom of the test tube for conventional coal tar and petroleum pitches, can be used as an indicator of overall mesophase quality. Unfortunately, gas evolution from all of the small-scale extracts was so copious and disruptive that no well-defined coalesced mesophase formed at the bottom of the test tube. The actual appearance of all of the residues from test-tube heat treatments of the extracts is shown schematically in Figure 19. Because of the effects of gas evolution, we were not able to obtain a definitive evaluation of the uniformity and quality of the mesophase formed in the small-scale extract by the test-tube method. However, based on appearance, the overall mesophase quality was judged to be borderline for a needle coke precursor. The true mesophase quality may have been somewhat better, but the effects from the gas evolution precluded obtaining a definitive answer.

Finely divided solids were detected around the perimeter of most of the coalesced mesophase regions and many of the larger mesophase spheres in all of the test-tube residues. These solids provided a local, short-range decrease in the mesophase domain size, but the effect on the overall quality was minor.

Qualitative SEM/EDX analyses were carried out on the finely divided solids in the C-296-2 (Thermal, 450°C - 1-hour) extract. Most appeared to be carbonaceous, some contained high Si, Al, O levels ( $\text{SiO}_2 + \text{Al}_2\text{O}_3$ ?), and some contained high Cu, Fe, S, and O levels (possibly a mixed copper and iron sulfate). The WVU Group suggested that the high Cu content in the last type of solid may have originated via attack of a copper gasket by the hot reactant mixture in the small-scale apparatus.

**Table III**  
**Summary of Hot-Stage Microscopy Observations on 450°C - 1-Hour Samples**

SAMPLE	INITIAL SOFTENING TEMP., °C	ONSET OF VIGOROUS BUBBLING, °C	INITIAL DETECTION OF MESOPHASE, TIME AT 400°C
C269-2 Thermal	198	255-275	21 min.
C279-1 CF-1, Fe <sub>2</sub> S <sub>3</sub>	155	250	16
C288-2 CF-2, Mo(S)	125	235	24

B. Large-Scale Extracts from Hydrogenated Coal

1. Sample Preparation

Following our evaluation of the small-scale extracts, we recommended that the 450°C hydrotreated coal materials be scaled up for evaluation by the Team. The extracts produced without the use of any catalyst appeared suitable for testing as a binder and impregnating pitch and as a precursor for needle coke and mesophase pitch for fibers.

Scaleup of the hydrotreated coal extracts was performed at WVU by hydrotreating 600 grams of powdered coal in 1-gallon batch reactors. They investigated 2 different coals using hydrotreatment times of 1 and 2 hours at 450°C. A description of the 4 materials produced (A066, A076, A075, and A073) and the properties measured at WVU are summarized in Table IV. The materials were also vacuum dried for an extended time in order to remove all of the retained NMP. We received about 200 gms of each material for further characterization.

**Table IV**  
**Large-Scale, Hydrotreated Coal Extracts**

Prepared Conditions	A066 Coal 13421 450°C - 1 hr.	A076 Coal 13421 450°C - 2 hr.	A075 Coal 13423 450°C - 1 hr.	A073 Coal 13423 450°C - 2 hr.
SP, °C	155.8	101.7	129.3	112.5
Density, g/cc	1.264	1.227	1.220	1.223
TI, Wt %	22.3	12.1	19.8	20.9
Coke Yield, Wt %	62.4	54.0	54.7	52.6

2. Analytical Data for Large-Scale 450°C Hydrotreated Coal Extracts

The data and properties obtained for the four large-scale coal extracts are summarized in Table V. We fully characterized the more extensively hydrotreated (2-hour) samples because these had the lowest softening points, making them more suitable for use as binder or impregnating pitch. In general, these larger scale products were superior in behavior to the small-scale samples discussed previously. One major characteristic was the essentially complete absence of any retained NMP. The N and O contents were therefore substantially reduced, but they were still much higher than those found in conventional coal tar pitches. The C/H ratios are similar to those in the comparably treated small-scale extracts.

Only the 2-hour products had softening points in the range of those for conventional carbon binder and impregnant pitches. The carbon yields of 52% for these materials are similar to those for commercial solids-free pitches of comparable softening point.

**Table V**  
**Properties of Large-Scale WVU Coal Extracts**

	<b>A076</b> 450°C - 2 Hrs. Coal 13421	<b>A066</b> 450°C - 1 Hr. Coal 13421	<b>A073</b> 450°C - 2 Hrs. Coal 13423	<b>A075</b> 450°C - 1 Hr. Coal 13423
SP, °C	104.7	160.7	115.3	134.5
MCC %	52.9	63.0	51.7	53.7
TGA Yd%	34	45	31	36
C, %	88.9	-----	89.6	-----
H, %	5.85	-----	5.78	-----
C/H Ratio	1.28	-----	1.30	-----
N, %	2.20	-----	1.90	-----
O, %	2.74	2.27	2.49	3.33
S, %	0.45	-----	0.33	-----
Ar H %	45	44	48	39
Mn (GPC)	367	417	389	388
Mesophase (μ)	272	166	136	78

The proton NMR spectra for the four materials in Table V are presented in Figures 20-23. All the extracts show substantial contents of hydrogenated ring systems. The decrease in the long chain aliphatic hydrogen peak at about 1.3 ppm, with extended hydrogenation, is particularly noticeable for the WVGS-13423 coal. The TGA curves for these large-scale extracts are shown in Figures 24-27. It is evident that the improved sample-drying procedure has removed any retained NMP. As expected, the total TGA weight losses increase for the more severely hydrotreated samples. The GPC curves for the four materials shown in Figures 28-31 do not exhibit any negative peaks due to NMP. The more severely hydrotreated coals show a broader molecular weight distribution (MWD) and the presence of an unresolved component at 280 a.m.u. when compared to the 1-hour samples.

### 3. Characterization of Extract Distillation Residues

We also received 3 distillation residues prepared by vacuum stripping of Sample A073 at temperatures of 250°C, 350°C, and 400°C. The materials are described in Table VI. The samples were prepared to determine whether we could detect any chemical changes resulting from heat treating at these temperatures.

**Table VI**  
**Vacuum Distillation Residues from Sample A073 (WVGS 13423 - 450°C - 2 Hours)**

SAMPLE	DISTILLATION TEMP., °C	YIELD, %
D035-1	250	76
D035-2	350	70
D035-3	400	68

We obtained NMR and TGA data for these residues and the results are summarized in Table VII. The NMR results show an increasing aromaticity with increasing depth of distillation. It is not possible to determine whether this effect is due to removal of volatile hydrogenated components during distillation or to low-temperature cracking reactions. The NMR data for the distillation residues are presented in Figures 32-34 while the TGA data are shown in Figures 35-37.

**Table VII**  
**Characterization of Hydrotreated Coal Extract Distillation Residues**

SAMPLE	% AROM H	% LONG CHAIN*	TGA Yd. %
D035-1	52	21	43
D035-2	55	22	58
D035-3	60	15	57

\* % of aliphatic H that is long chain

4. Mesophase Evaluations of Large-Scale Extracts

Hot-stage microscopy examinations were carried out on both of the 450°C - 2-hour extracts in the same run. The A073 extract began to soften visibly at 94°C, and the A076 extract had slumped noticeably by 104°C while they were being heated at 6°C/min. to 400°C. The low initial visible softening temperatures on the hot-stage are consistent with the low Mettler S. P.'s given in Tables IV and V for these two extracts.

Both of these large-scale extracts evolved much less gas between 250°C -350°C as compared to the levels seen with all of the small-scale extracts. They behaved much more like conventional petroleum and coal tar pitches in their low-temperature gas evolution characteristics. Apparently, the combined effects of more stringent drying conditions to remove residual NMP and the somewhat more severe reaction conditions



employed by the WVU Group for the large-scale extracts had nearly eliminated the severe gas evolution problem detected with the small-scale extracts.

Small amounts of mesophase spheres were detected at the edges of the cover glass sandwiches in both A073 and A076 extracts after ~25 min. at 400°C, indicating that they had comparable initial mesophase formation rates. These onset times are only marginally longer than those noted for the 450°C - 1-hour, small-scale samples.

A hot-stage microscopy run carried out with both of the 450°C - 1-hour samples showed an initial softening temperature of 114°C for A066 extract and 146°C for A075 extract. These results are reasonably consistent with the Mettler S. P. data in Tables IV and V.

The A066 and A075 extracts also evolved much less gas in the 250°-350°C range than the small-scale extracts. Clearly, the beneficial effects from the more stringent drying conditions and the somewhat different reaction conditions employed by the WVU Group were realized after only 1 hour at a nominal reaction temperature of 450°C.

Small amounts of mesophase were detected at the perimeter of the cover glass sandwiches for both A066 and A075 extracts after 21-22 min. at 400°C on the hot-stage microscope. Hence, the mesophase formation rates of these large-scale 450°C - 1-hour samples were comparable to each other and to those for both the 450°C - 1 hour large-scale and small-scale extracts. Both of the 450°C - 1-hour samples appeared to generate slightly more true pyrolysis gas during the hold period at 400°C than the 450°C - 2-hour samples. This is an indication that the 450°C - 1-hour samples had a slightly higher initial rate for cracking reactions.

All of the large-scale extracts were heat treated in boats at 400°C to produce approximately 50% mesophase and then subjected to the "annealing" process for measurement of mesophase domain size. Polarized light (crossed polars) micrographs taken at 50X and 250X magnification of the derived mesophase are shown in Figures 38-41. It is apparent that the more extensively hydrotreated extracts give larger domain size mesophase than the less-treated 1-hour samples. It is also evident that the mesophase derived from the WVGS-13421 coal extract has a larger domain size than that from WVGS-13423 coal for the same hydrotreatment severity. The A076 extract from WVGS coal 13421 hydrotreated 2 hours at 450°C gave the largest mesophase domain size of any of the large-scale extracts. Mesophase domain sizes were obtained by image analysis and are reported in Table V. It is apparent that the domain sizes of the extracts from the

WVGS-13423 coal are significantly smaller than the corresponding extract from the WVGS-13421 coal. The measured domain size of 272 $\mu$ m for the A076 extract is in the range found for excellent needle coke precursors.

The presence of solid particles in the coalesced mesophase and at the mesophase/isotropic phase boundary was noted to some extent in the annealed samples from all of the large-scale extracts. The WVGS-13421 extracts had more of this finely divided material than those derived from WVGS-13423. Figure 42 shows an area where the solids have collected or segregated in a small region near the edge of the coalesced mesophase from the A076 extract sample from WVGS-13421. SEM/EDX analyses of the particles in this cluster showed that several different materials were present. Carbonaceous particles with very high C and modest S levels were numerous, presumably coke-like material from the original coal which had not reacted during hydrotreatment. Particles with high Si, Al, and O contents were also prevalent, presumably very small ash particles from the original coal which had not been removed completely during the filtration step after hydrotreatment and NMP extraction. There was also a significant number of particles with high Fe, Cr, S, and possibly O contents, presumably iron and chromium sulfates. A few particles with high Ti and O contents (TiO<sub>2</sub>?) or high Cu, K, and small O contents (calcium and potassium sulfides or sulfates?) were also detected. As expected, most of these finely divided solids originate in the coal. Presumably, their removal would be more complete in larger scale equipment.

### C. Additional Extract Samples

Small quantities of three additional extract materials were received from the WVU Group toward the end of the contract period. Two of the samples were the toluene soluble (TS) portions from hydrotreated coal extracts. D065-2 was the TS portion from a 450°C - 1-hour hydrotreatment of the WVGS-13423 coal, and D054 was TS from a 450°C - 2-hour hydrotreatment of WVGS-13421 coal. Both of these extracts melted in the 90°-100°C range on the hot-stage microscope and had low yields after heating to 400°C at 6°C/min. Both were also very refractory during the hold period at 400°C and required 1.5-2.0 hours to produce detectable mesophase spheres. An attempt to evaluate their mesophase domain size was unsuccessful because these TS materials were so unreactive under the normal heat treatment conditions and had low yields. The low softening point behavior and low yield upon heat treatment are consistent with the low MW range encompassed by toluene soluble material.

A third extract (A122) was prepared at WVU by carrying out a 450°C - 1-hour hydrotreatment on the toluene insoluble (TI) fraction from a large-scale NMP extract from an initial 450°C - 2-hour hydrotreatment of WVGS-13423 coal. This double-hydrotreated material had an initial softening temperature of 188°C and formed mesophase spheres quite rapidly at 400°C, probably due to distillation of volatiles. Additional mesophase formed at a normal rate for

thermal processing of good feedstocks at 400°C. The mesophase quality appeared excellent on the hot stage. A standard mesophase evaluation showed that A122 had a domain size of approximately 200µm by visual observation on the annealed sample. Quantitative image analysis was not reliable on this sample because the mesophase in the annealed specimen had so many cracks. The oxygen content of the double-hydrotreated A122 material was still 1.88%, indicating that the O heteroatom structures in these coal extracts are very resistant to removal by hydrogenation. Even with this high oxygen content, the A122 extract still formed a high-quality mesophase.

#### D. Activation of Coal Extraction Residues

The economics of the coal extraction process would be improved if the extraction residues also had some value. For this purpose, we attempted to activate these materials to produce a high surface area carbon. We obtained samples of the NMP extraction residue of raw coal WVGS-13421, as well as the extraction residue of a coal hydrotreated at 450°C for 2 hours (A-67).

Both materials were activated in a H<sub>2</sub>O/N<sub>2</sub> mixture at 900°C using a tumbling furnace. The hydrotreated (A-67) residue was very refractory and after about 81 hours of treatment gave a 64% burn-off. The raw coal residue was much more reactive and gave about 85% burn-off after 48 hours.

The surface areas and pore characteristics were determined for both materials at the Oak Ridge Laboratory by T. Burchell and E. L. Fuller. The measured surface area for the activated residue from the untreated coal was 770 m<sup>2</sup>/gm while that for the hydrotreated coal was 212 m<sup>2</sup>/gm. The lower surface area and refractory nature of the hydrotreated residue is expected since it would have a greater ash content than the residue from the untreated coal. Since hydrotreatment increases the extraction yield, it would also lead to a higher ash content in the insoluble residue.

In a subsequent study, we obtained a second sample of the extraction residue from untreated coal and attempted to produce active carbons using different amounts of burn-off. We prepared 3 materials with active carbon yields of 50, 65, and 68%. These samples were also submitted to T. Burchell for surface area measurement. The results are summarized in Table VIII along with our measured ash analysis for these activated carbons. The results show that surface area does increase with increasing burn-off. However, more detailed studies would be needed to determine the optimum activation that can be achieved for these residues.

**Table VIII**  
**Active Carbons from Coal Extraction Residue (Activated at 900°C, H<sub>2</sub>O)**

SAMPLE	ACTIVE CARBON Yd. %	SURFACE AREA m <sup>2</sup> /gm	% ASH
65-1	50	464	4.6
65-3	65	410	4.3
65-2	68	332	4.2

**E. Fabrication of Graphite Artifacts from Hydrotreated Coal Extracts**

We produced a graphite artifact using coal extract-derived coke as the filler and the coal extract itself as the binder. The coal extract coke was produced from WVGS-13421 coal hydrotreated for 450°C for 2 hours. The raw coke was prepared at WVU and then calcined to 1,100°C by T. Burchell at the Oak Ridge Laboratory. The same coal NMP extract was used as the binder for graphite fabrication. The extract (D-085) had a 113°C Mettler softening point and a 52.6% MCC value. After milling, the calcined coke was mixed with 1/3 by weight of the coal extract binder and then extruded to produce 19mm-diameter rods. The rods were baked and graphitized to 3,000°C. The properties measured for the graphitized rods were the following:

Coefficient of Thermal Expansion (CTE) =  $0.26 \times 10^{-6}/^{\circ}\text{C}$   
 Specific Resistance = 9.55 micro-ohm-meters  
 Density = 1.376 g/cc

The very low CTE value of 0.26 is similar to that measured for electrode graphite derived from petroleum needle coke. The relatively low density for the fabricated artifact could be due to an inappropriate binder level or to a puffing effect from the nitrogen in the coke.

**V. DISCUSSION**

Our screening evaluation studies have demonstrated that the coal extraction process has the potential for producing acceptable products for all the applications anticipated for the program. However, hydrotreatment is necessary to produce materials that are suitable for use as pitches, coke precursors, or mesophase raw materials.

The extract from the hydrotreated coal WVGS-13421, 450° - 2 hours appears to be acceptable as either a carbon binder or impregnation pitch. Summarized in Table IX is a comparison of the properties for the A076 extract with those for commercial coal tar binder and petroleum impregnant pitches. The carbon yield (MCC) for the extract is quite high for a solids-free pitch with a softening point of 105°C. Some of the analytical characteristics of the extract are quite unusual in comparison to those for the commercial pitches. The low aromatic

hydrogen content reflects the effects of hydrotreatment of the precursor coal. The high N and O contents are also unusual for a binder or impregnant pitch material.

Our mesophase evaluation studies show that a material, such as the NMP extract of the WVGS-13421 coal hydrotreated for 450°C for 2 hours, is capable of forming a very large domain mesophase. This extract could therefore be used to produce a mesophase pitch which may be acceptable for fibers. The mesophase quality is comparable to that for precursors of needle coke. The high nitrogen content would be a drawback for processing of such a coke. The excellent mesophase development for a pitch such as the A076 extract is surprising because of its very high oxygen content. A high oxygen content in coal conversion materials has been previously associated with a high reactivity and a poor mesophase quality. Obviously, the form of oxygen in these hydrotreated coal extracts is different from that in typical coal conversion products and does not lead to a fast coking reactivity.

Our results have shown that a hydrotreated coal extract such as the A076 can be used directly as a binder pitch without any subsequent processing. The direct production of pitches by the extraction process could have advantages over the current commercial coal tar binder pitch processes which involve the distillation of coal tar. The need for coke oven processing for tar generation and the handling of by-products from tar distillation would be avoided. It is likely that pitches with different properties could be obtained by controlling the hydrotreatment and extraction conditions.

We have demonstrated that coke derived from hydrotreated coal extract can be used to produce graphite with a low coefficient of thermal expansion, a requisite for graphite electrodes. By altering the processing conditions and the use of blends, it would be possible to produce graphites with varying degrees of anisotropy. We had shown previously that isotropic graphite can be produced from the coal extract<sup>(1)</sup>. No other process has the flexibility for producing this range of carbon materials. The high nitrogen contents of the extracts could, however, be a drawback for the use of these materials for some graphite products.

Finally, the extraction residue can be activated to produce an activated carbon. The best procedure for achieving an optimum active carbon still needs to be defined. However, the use of the residue for a commercial applications would definitely improve total process economics.

**Table XI**  
**Properties of Coal Extract and Commercial Pitches**

	<b>EXTRACT A076</b>	<b>COAL BINDER</b>	<b>PETROLEUM IMPREGNANT</b>
SP, °C	105	112	120
MCC %	53	58	52
C, %	88.9	93.8	91.5
H, %	5.85	3.92	5.0
N, %	2.20	0.70	0.6
O, %	2.74	0.93	0.5
S, %	0.45	0.56	2.5
ArH %	45	85	55
Mol Wt.	370	410	500

References:

- (1) "Coal-Based Nuclear Graphites for the New Production Gas-Cooled Reactor" DOE Grant DE FG02-91 NP00159, Final Report, May 31, 1994
- (2) R. A. Greinke and L. H. O'Connor, *Anal. Chem.* 52, 1877 (1980)
- (3) R. T. Lewis, I. C. Lewis, R. A. Greinke, S. L. Strong, *Carbon* 25, 289 (1987)
- (4) R. T. Lewis, Extended Abstract for 12<sup>th</sup> Biennial Carbon Conference, Pittsburgh, PA, p. 215, 1975
- (5) E. L. Fuller, ORNL, personal communication

Figures

- Figure 1: Proton NMR in CS<sub>2</sub> Solution for C-269-2, Hydrotreated Coal 450°C - 1 Hour, No Catalyst
- Figure 2: Proton NMR in CS<sub>2</sub> Solution for C-279-1, Hydrotreated Coal 450°C - 1 Hour, Fe<sub>2</sub>S<sub>3</sub> Catalyst
- Figure 3: Proton NMR in CS<sub>2</sub> Solution for C-288-2, Hydrotreated Coal 450°C - 1 Hour, Mo(S) Catalyst
- Figure 4: TGA Curve for C-269-2, Hydrotreated Coal 450°C - 1 Hour, No Catalyst
- Figure 5: TGA Curve for C-279-1, Hydrotreated Coal 450°C - 1 Hour, Fe<sub>2</sub>S<sub>3</sub> Catalyst
- Figure 6: TGA Curve for C-288-2, Hydrotreated Coal 450°C - 1 Hour, Mo(S) Catalyst
- Figure 7: Gel Permeation Chromatography (GPC) Curve for C-269-2, Hydrotreated Coal 450°C - 1 Hour, No Catalyst
- Figure 8: Gel Permeation Chromatography (GPC) Curve for C-279-1, Hydrotreated Coal 450°C - 1 Hour, Fe<sub>2</sub>S<sub>3</sub> Catalyst
- Figure 9: Gel Permeation Chromatography (GPC) Curve for C-288-2, Hydrotreated Coal 450°C - 1 Hour, Mo(S) Catalyst
- Figure 10: Proton NMR in CS<sub>2</sub> for C-300-3, Hydrotreated Coal 450°C - 2 Hours, No Catalyst

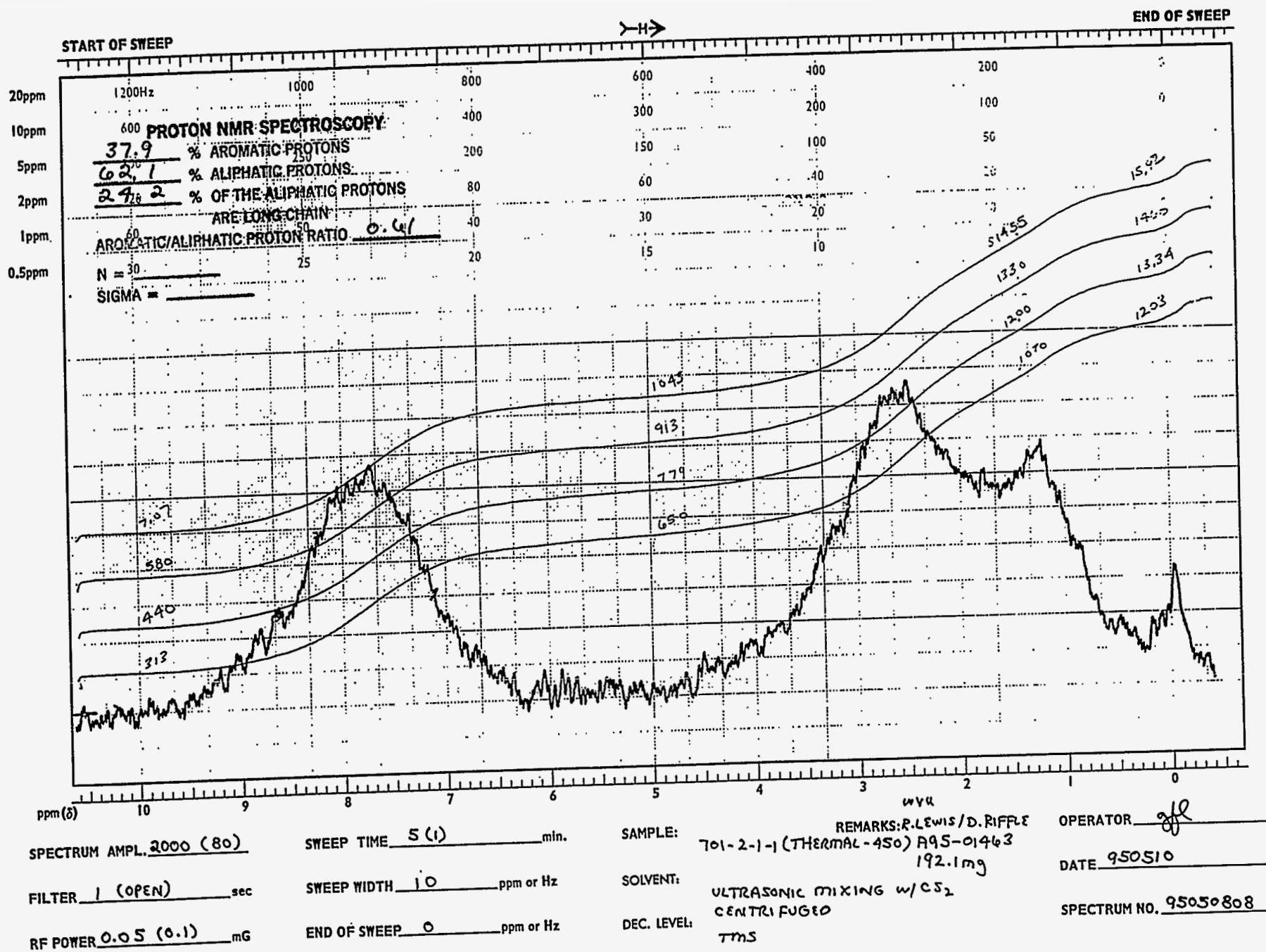
- Figure 11: Proton NMR in CS<sub>2</sub> for C-300-6, Hydrotreated Coal 450°C - 2 Hours, Fe<sub>2</sub>S<sub>3</sub> Catalyst
- Figure 12: Proton NMR in CS<sub>2</sub> for C-300-9, Hydrotreated Coal 450°C - 2 Hours, Mo(S) Catalyst
- Figure 13: TGA Curve for C-300-3, Hydrotreated Coal 450°C - 2 Hours, No Catalyst
- Figure 14: TGA Curve for C-300-6, Hydrotreated Coal 450°C - 2 Hours, Fe<sub>2</sub>S<sub>3</sub> Catalyst
- Figure 15: TGA Curve for C-300-9, Hydrotreated Coal 450°C - 2 Hours, Mo(S) Catalyst
- Figure 16: Gel Permeation Chromatography Curve for C-300-3, Hydrotreated Coal 450°C - 2 Hours, No Catalyst
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- Figure 26: TGA Curve for A073 from Hydrotreated Coal WVGS-13423 - 450°C, 2 Hours
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Figure 1: Proton NMR in CS<sub>2</sub> Solution for C-269-2, Hydrotreated Coal  
450°C - 1 Hour, No Catalyst

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WCV-380-K10-2

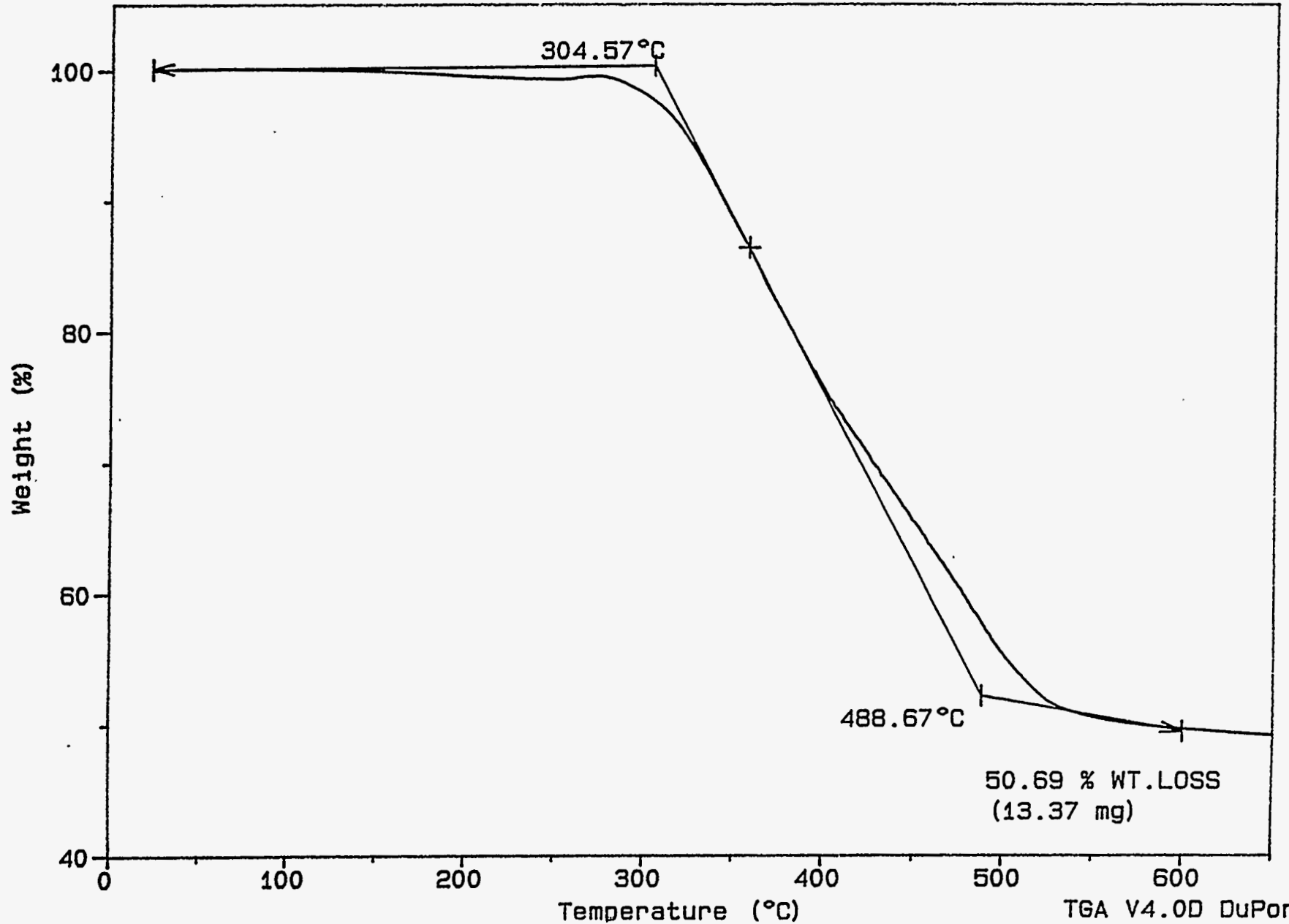


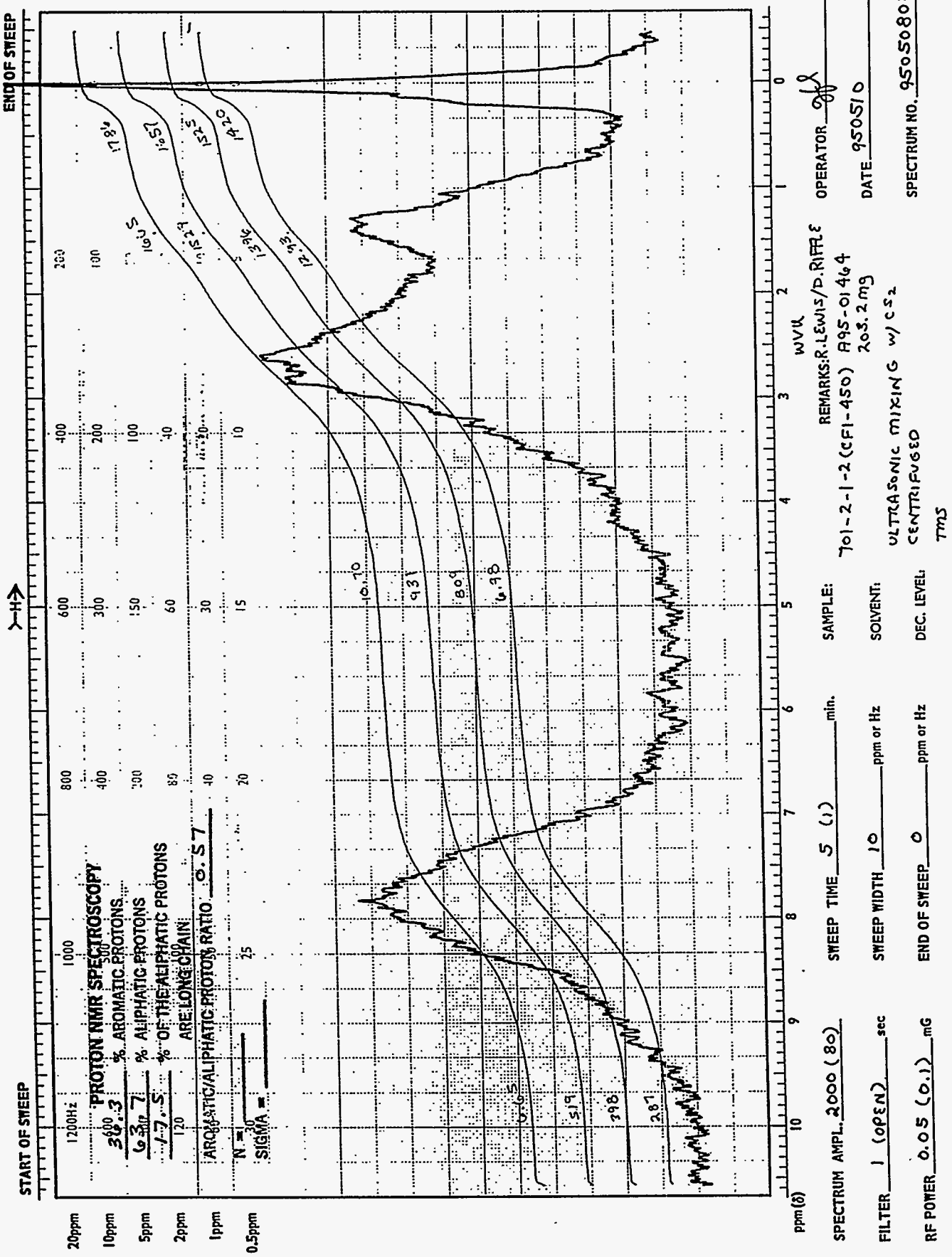
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Comment: ARGON 100CC/MIN Pt PAN

TGA

File: 95050808A.08  
Operator: gfl  
Run Date: 18-May-95 16:23

Figure 4: TGA Curve for C-269-2, Hydrotreated Coal 450°C - 1 Hour, No Catalyst





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Figure 2: Proton NMR in CS<sub>2</sub> Solution for C-279-1, Hydrotreated Coal 450°C - 1 Hour, Fe<sub>2</sub>S<sub>3</sub> Catalyst

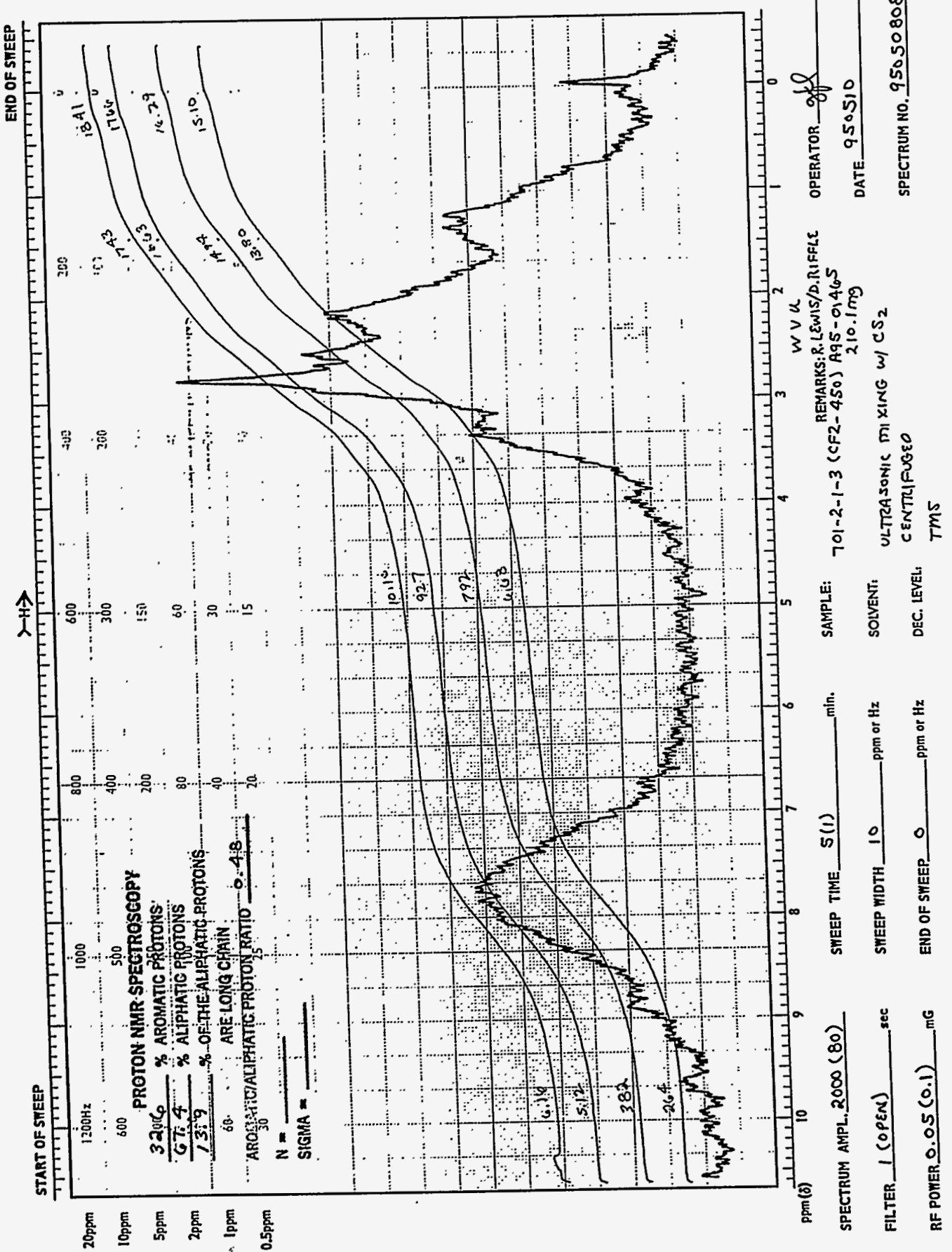


Figure 3: Proton NMR in CS<sub>2</sub> Solution for C-288-2, Hydrotreated Coal 450°C - 1 Hour, Mo(S) Catalyst

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W-100-1178

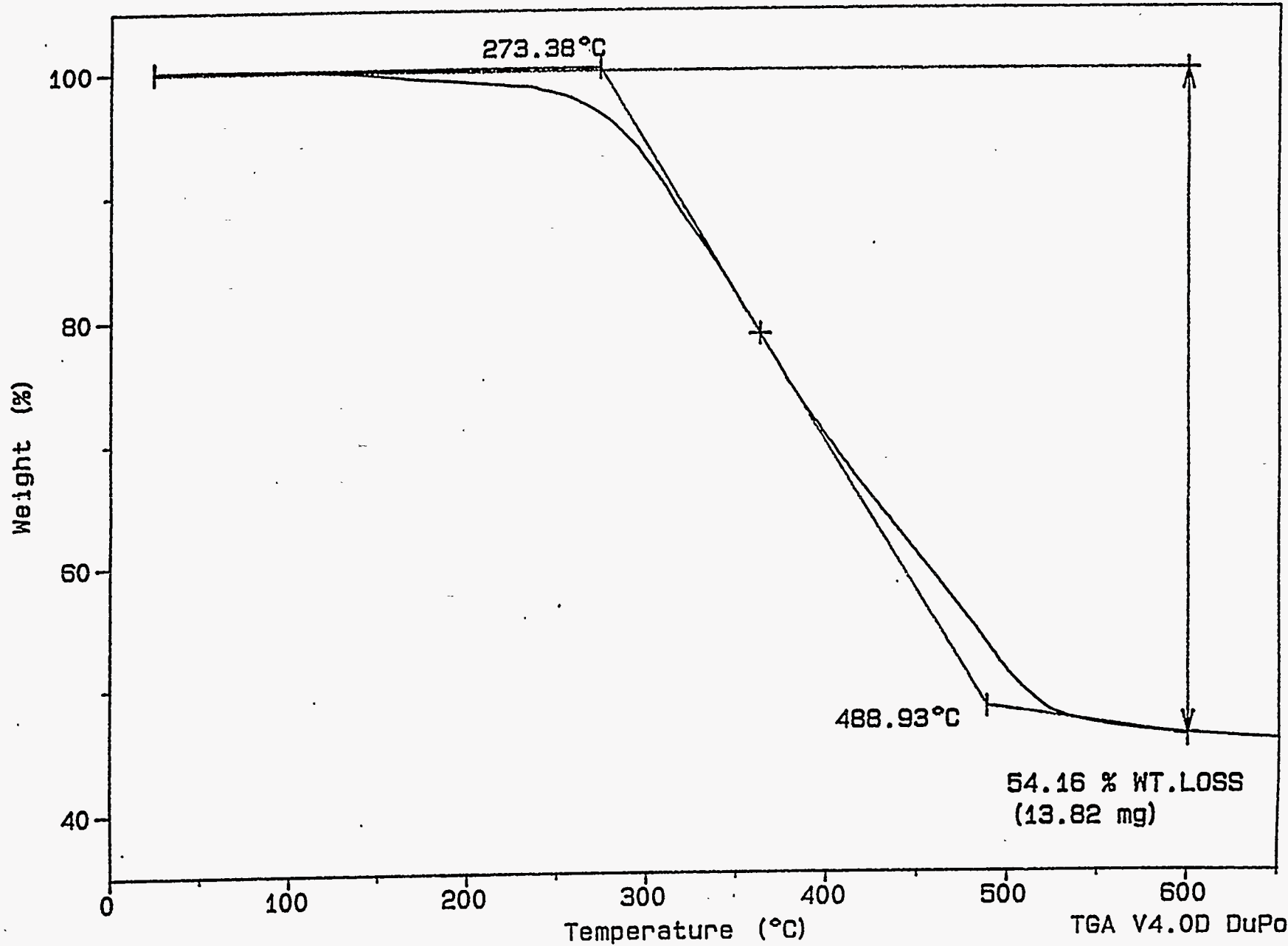
M  
WCV-360-K10-2

Sample: 701-2-1-2 (CF1-450)  
Size: 25.5111 mg  
Method: 10C/MIN  
Comment: ARGON 100CC/MIN Pt PAN

A95-01464 TGA

File: 95050808B.09  
Operator: gfl  
Run Date: 18-May-95 13:48

Figure 5: TGA Curve for C-279-1, Hydrotreated Coal450°C - 1 Hour, Fe2S3 Catalyst



Sample: 701-2-1-3 (CF2-450)

A95-01465

TGA

File: 95050808C.05

Size: 24.2128 mg

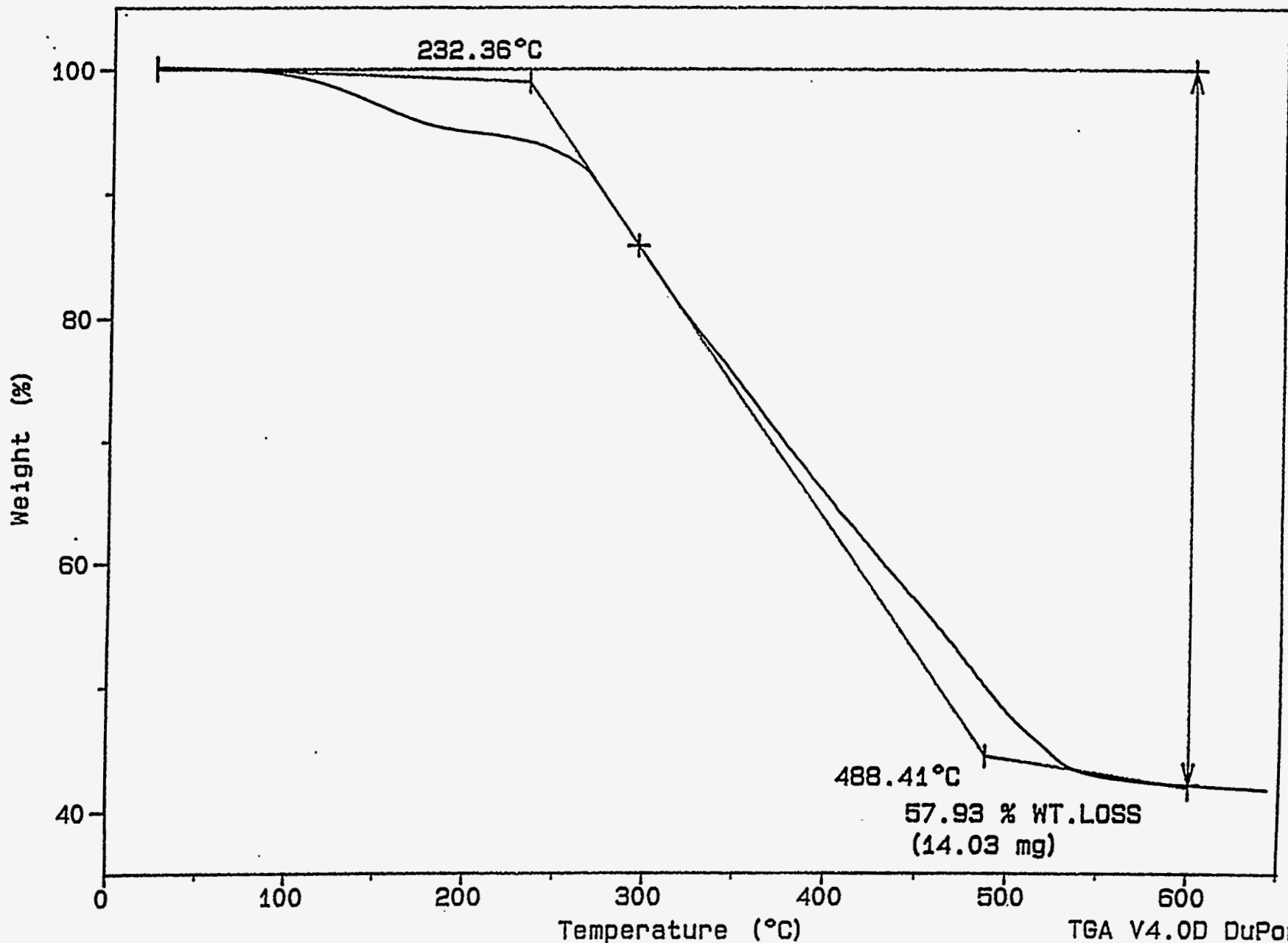
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Method: 10C/MIN

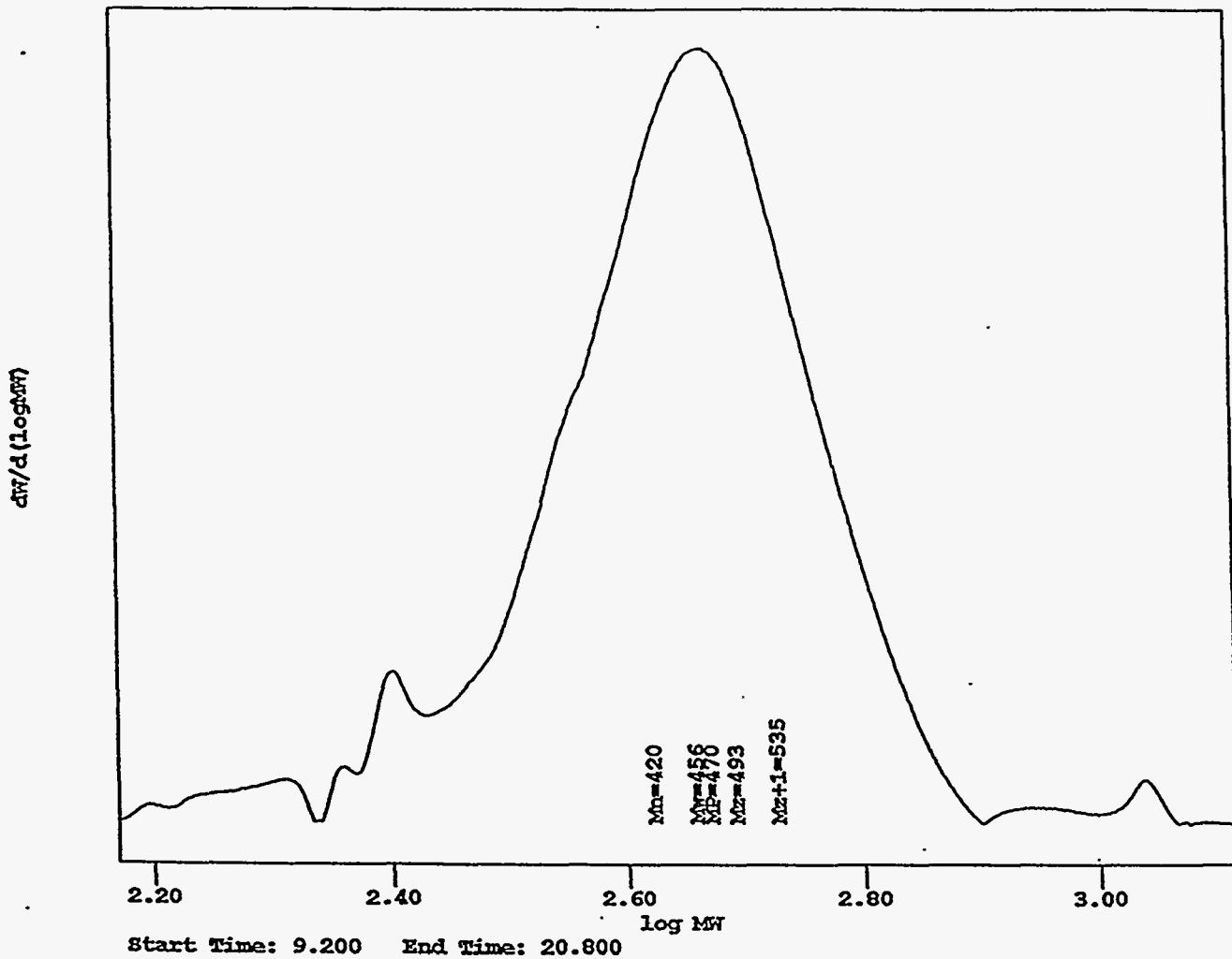
Run Date: 12-May-95 15:11

Comment: ARGON 100CC/MIN Pt PAN

Figure 6: TGA Curve for G-288-2, Hydrotreated Coal 450°C - 1 Hour, Mo(S) Catalyst



Project Name:	GPC	Sample Type:	Broad Unknown
Sample Name:	701-2-1-1	Volume:	100.00
Vial:	2	Run Time:	30.0 min
Injection:	1	Date Processed:	06/23/95 10:02:42 AM
Date Acquired:	06/22/95 01:55:58 PM	Pressure:	75
Processing Method:	GPC	Submitter:	hkm
Comments:	95050808: 1g3ml TCB Thermal - 450. prepped 5/16/95		



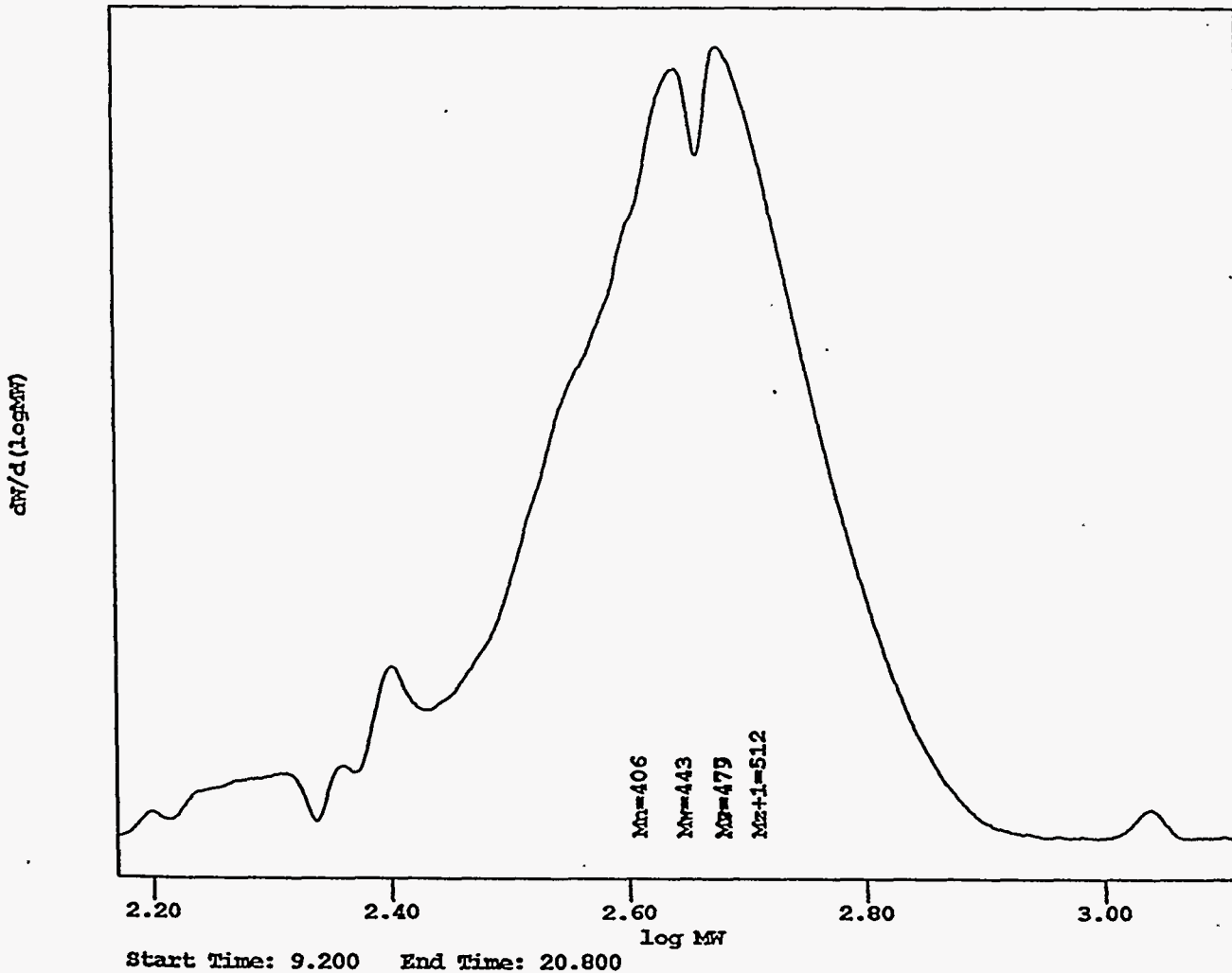
Peak Results

#	Ret Time (min)	Mn (Daltons)	MP (Daltons)	Mw (Daltons)	Mz (Daltons)	Mz+1 (Daltons)	Polydispersity
1	14.617	420	470	456	493	535	1.086609

Figure 7: Gel Permeation Chromatography (GPC) Curve for C-269-2, Hydrotreated Coal 450°C - 1 Hour, No Catalyst



Project Name:	GPC	Sample Type:	Broad Unknown
Sample Name:	701-2-1-2	Volume:	100.00
Vial:	3	Run Time:	30.0 min
Injection:	1	Date Processed:	06/23/95 10:02:13 AM
Date Acquired:	06/22/95 02:51:24 PM	Pressure:	75
Processing Method:	GPC	Submitter:	hkm
Comments:	95050808 .1g3ml TCB CF-1- 450 prepped 5/16/95		

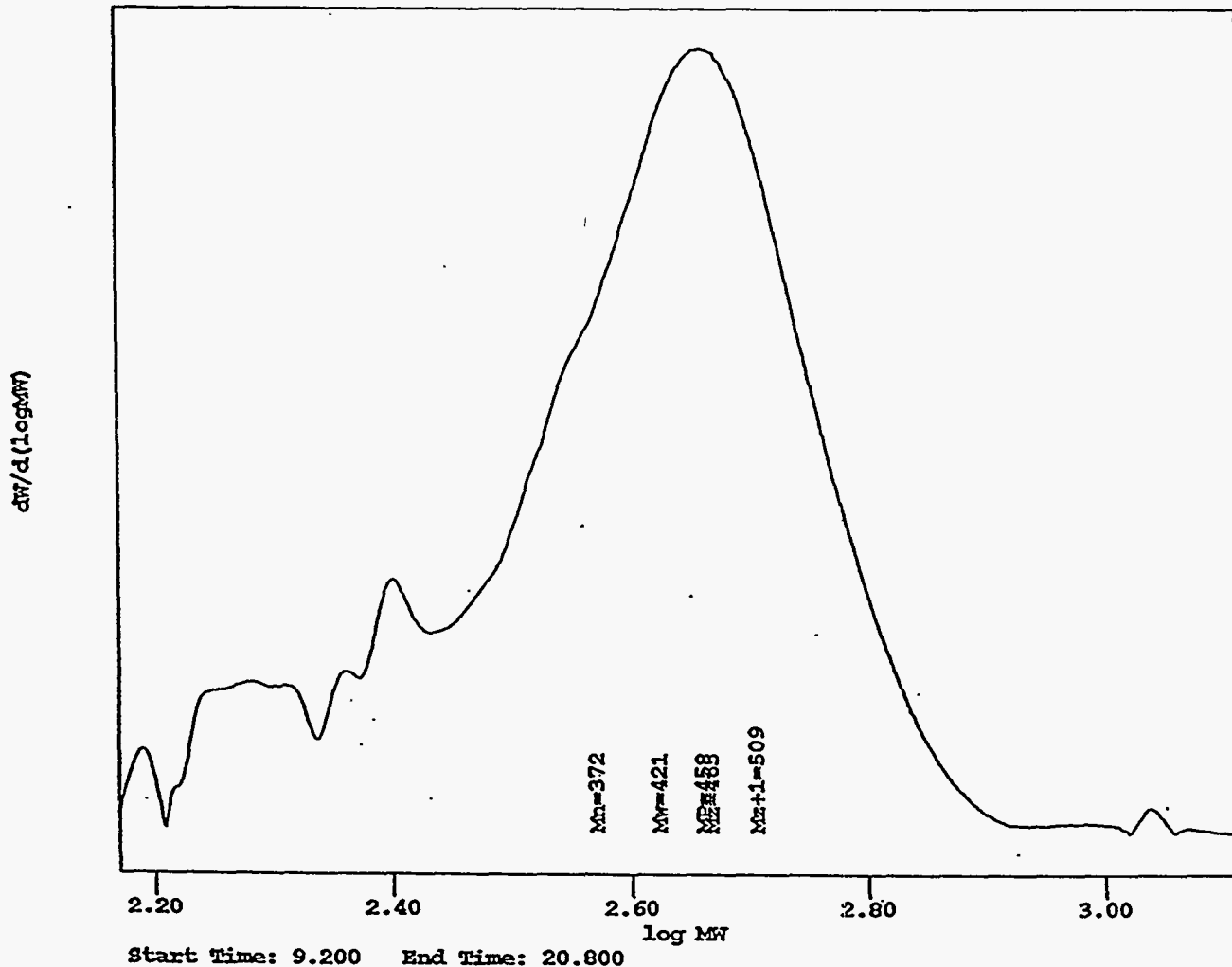


Peak Results

f	Ret Time (min)	Mn (Daltons)	MP (Daltons)	Mw (Daltons)	Mz (Daltons)	Mz+1 (Daltons)	Polydispersity
1	14.517	406	479	443	477	512	1.090000

Figure 8: Gel Permeation Chromatography (GPC) Curve for C-279-1, Hydrotreated Coal 450°C - 1 Hour, Fe<sub>2</sub>S<sub>3</sub> Catalyst

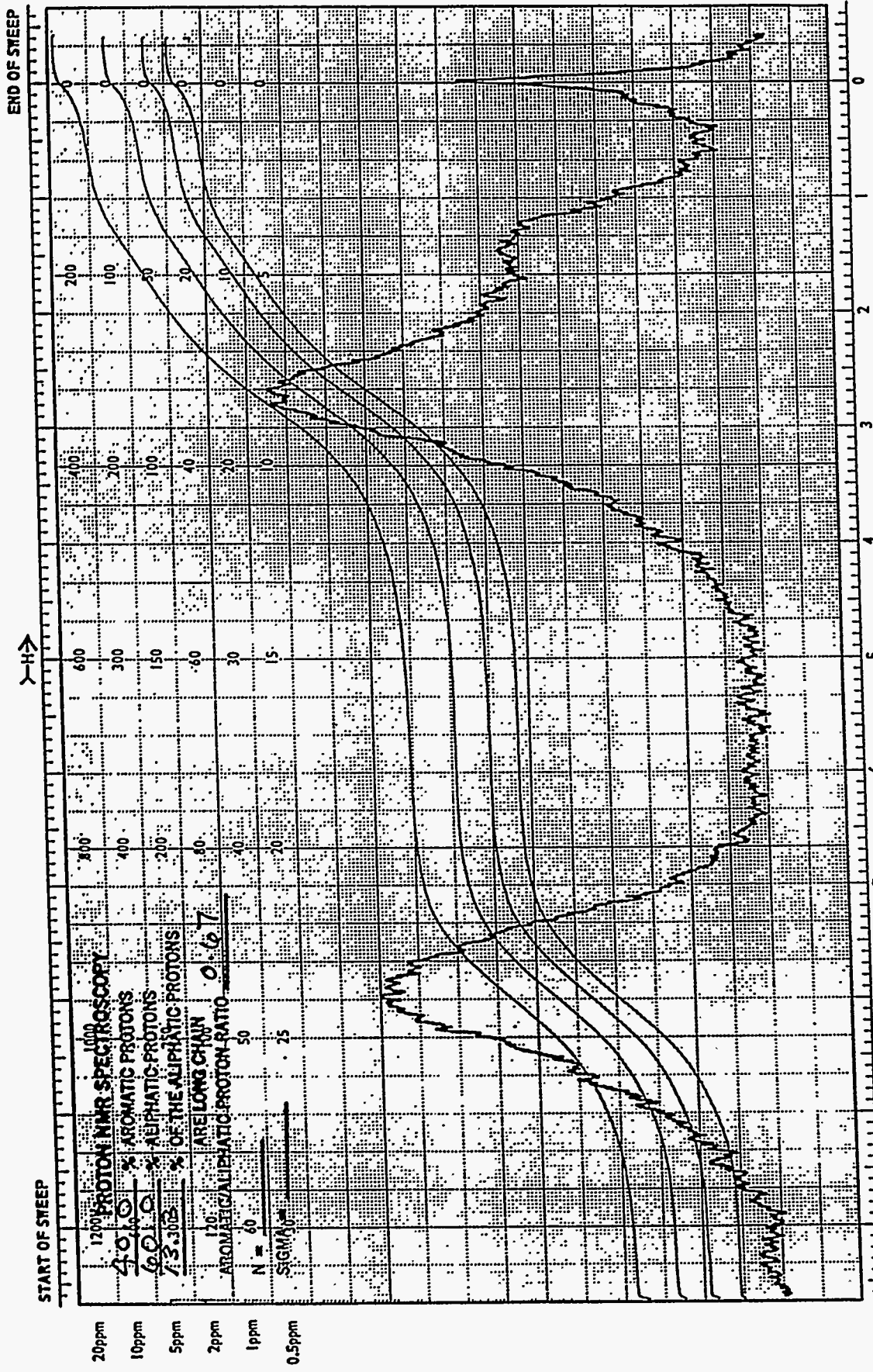
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Sample Name:	701-2-1-3	Volume:	100.00
Vial:	4	Run Time:	30.0 min
Injection:	1	Date Processed:	06/23/95 10:01:31 AM
Date Acquired:	06/22/95 03:32:41 PM	Pressure:	75
Processing Method:	GPC	Submitter:	hkm
Comments:	95050808 .1g3ml TCB CF-2- 450 prepped 5/16/95		



Peak Results

#	Ret Time (min)	Mn (Daltons)	MP (Daltons)	Mw (Daltons)	Mz (Daltons)	Mz+1 (Daltons)	Polydispersity
1	14.750	372	458	421	465	509	1.129627

Figure 9: Gel Permeation Chromatography (GPC) Curve for C-288-2, Hydrotreated Coal 450°C - 1 Hour, Mo(S) Catalyst

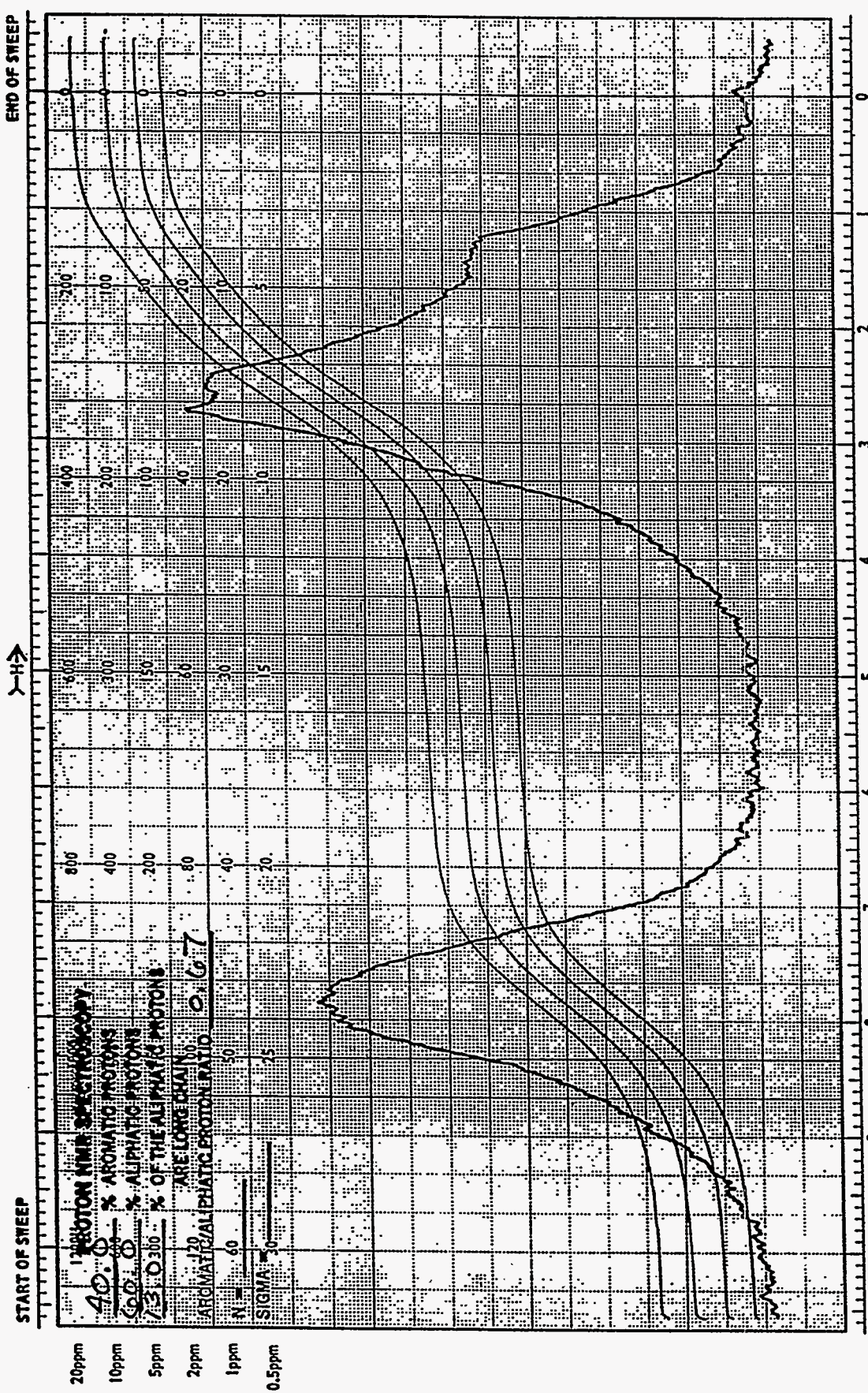


START OF SWEEP →      ← END OF SWEEP  
 1200 PROTON NMR SPECTROSCOPY  
 40.0 % AROMATIC PROTONS  
 60.0 % ALIPHATIC PROTONS  
 13.30% OF THE ALIPHATIC PROTONS  
 ARE LONG CHAIN  
 AROMATIC ALIPHATIC PROTON RATIO 0.67  
 N = 60  
 SIGMA 25  
 ppm (δ) 10 9 8 7 6 5 4 3 2 1 0

OPERATOR R. LEWIS  
 DATE 7-2-16-4 (A95-01823)  
 SPECTRUM NO. 95060801  
 REMARKS: REFLUXED IN CS<sub>2</sub>  
 TMS  
 SAMPLE: 701-2-16-4 (A95-01823)  
 SOLVENT: REFLUXED IN CS<sub>2</sub>  
 DEC. LEVEL: TMS  
 SPECTRUM AMPL. 2000 (85) SWEEP TIME 5 (1) min.  
 FILTER 1 (OPEN) sec SWEEP WIDTH 10 ppm or Hz  
 RF POWER 0.05 (0.1) mG END OF SWEEP 0 ppm or Hz

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Figure 10: Proton NMR in CS<sub>2</sub> for C-300-3, Hydrotreated. Coal 450°C - 2 Hours, No Catalyst



SPECTRUM AMPL. 1000 (25) SWEEP TIME 5 (1) min. SAMPLE: 701-2-6-8 (A95-01827)  
 FILTER 1 (OPEN) sec SWEEP WIDTH 10 ppm or Hz SOVENT: REFLUXED W/CS<sub>2</sub>  
 RF POWER 0.05 (.1) mG END OF SWEEP 0 ppm or Hz DEC. LEVEL: TMS

OPERATOR off DATE 950630 SPECTRUM NO. 95060801

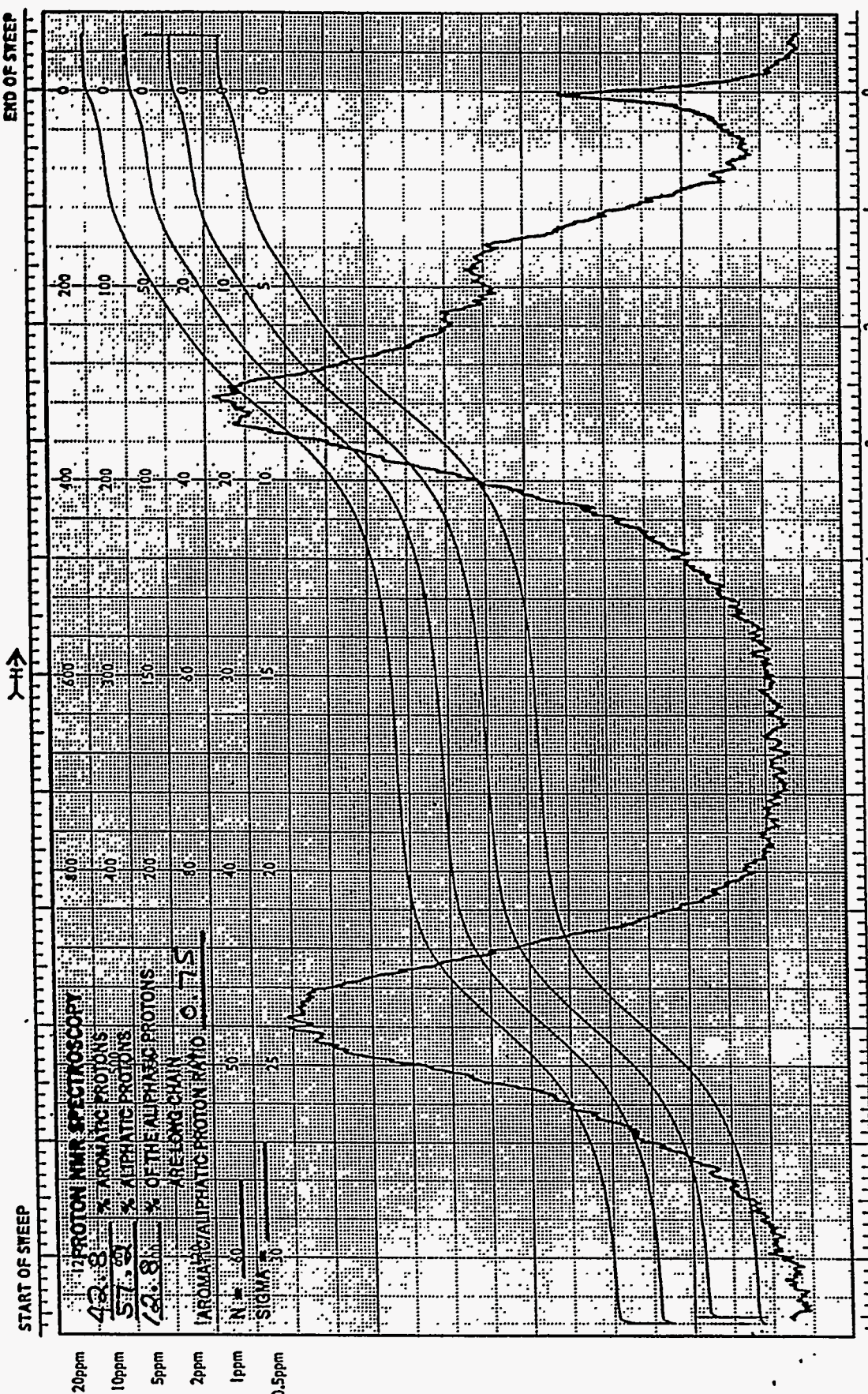
REMARKS: R. Lewis

20ppm  
 10ppm  
 5ppm  
 2ppm  
 1ppm  
 0.5ppm

1. PROTON NMR SPECTROSCOPY  
 40.0 % AROMATIC PROTONS  
 60.0 % ALIPHATIC PROTONS  
 73.0 % OF THE ALIPHATIC PROTONS  
 ARE LONG CHAIN  
 AROMATIC/ALIPHATIC PROTON RATIO 0.67  
 N 60  
 SIGMA 50



Figure 11: Proton NMR in CS<sub>2</sub> for C-300-6, Hydrotreated Coal 450°C - 2 Hours, Fe<sub>2</sub>S<sub>3</sub> Catalyst



12 PROTON NMR SPECTROSCOPY  
 48.8% AROMATIC PROTONS  
 57.2% ALIPHATIC PROTONS  
 72.8% OF THE ALIPHATIC PROTONS  
 ARE LONG-CHAIN  
 AROMATIC/ALIPHATIC PROTON RATIO 0.75  
 N = 60  
 SIGMA = 30

SPECTRUM AMPL. 1500 (40) min. OPERATOR: gfl  
 FILTER 1 (OPEN) sec. SAMPLE: 701-2-14-12 (A95-01825)  
 SWEEP WIDTH 10 ppm or Hz SOLENT: REFUXED w/CS<sub>2</sub>  
 END OF SWEEP 0 ppm or Hz DEC. LEVEL: 77MS  
 RF POWER 0.05 (0.1) mG SPECTRUM NO. 95060801

PERKINELMER  
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 BURLINGAME, CALIF. 94010  
 MADE IN U.S.A.

Figure 12: Proton NMR in CS<sub>2</sub> for C-300-9, Hydrotreated Coal 450°C - 2 Hours, Mo(S) Catalyst

C300-3

Sample: 701-2-16-4

A95-01823 TGA

File: 95060801A.01

Size: 24.8060 mg

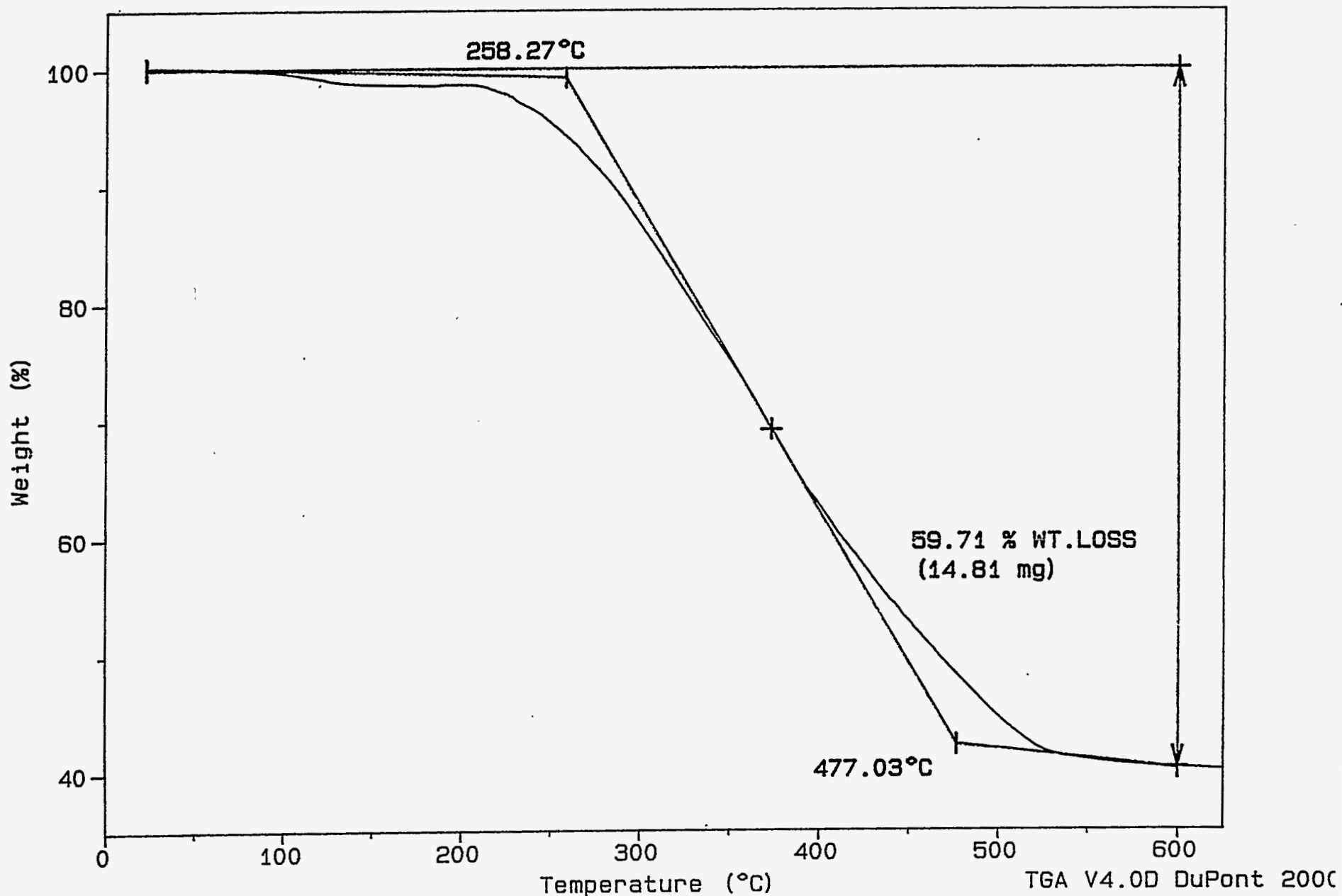
Operator: gfl

Method: 10C/MIN

Run Date: 23-Jun-95 11:28

Comment: ARGON 100CC/MIN Pt PAN

Figure 13: TGA Curve for C-300-3, Hydrotreated Coal  
450°C - 2 Hours, No Catalyst



C300-6

Sample: 701-2-16-8

A95-01824 TGA

File: 95060801B.01

Size: 24.4479 mg

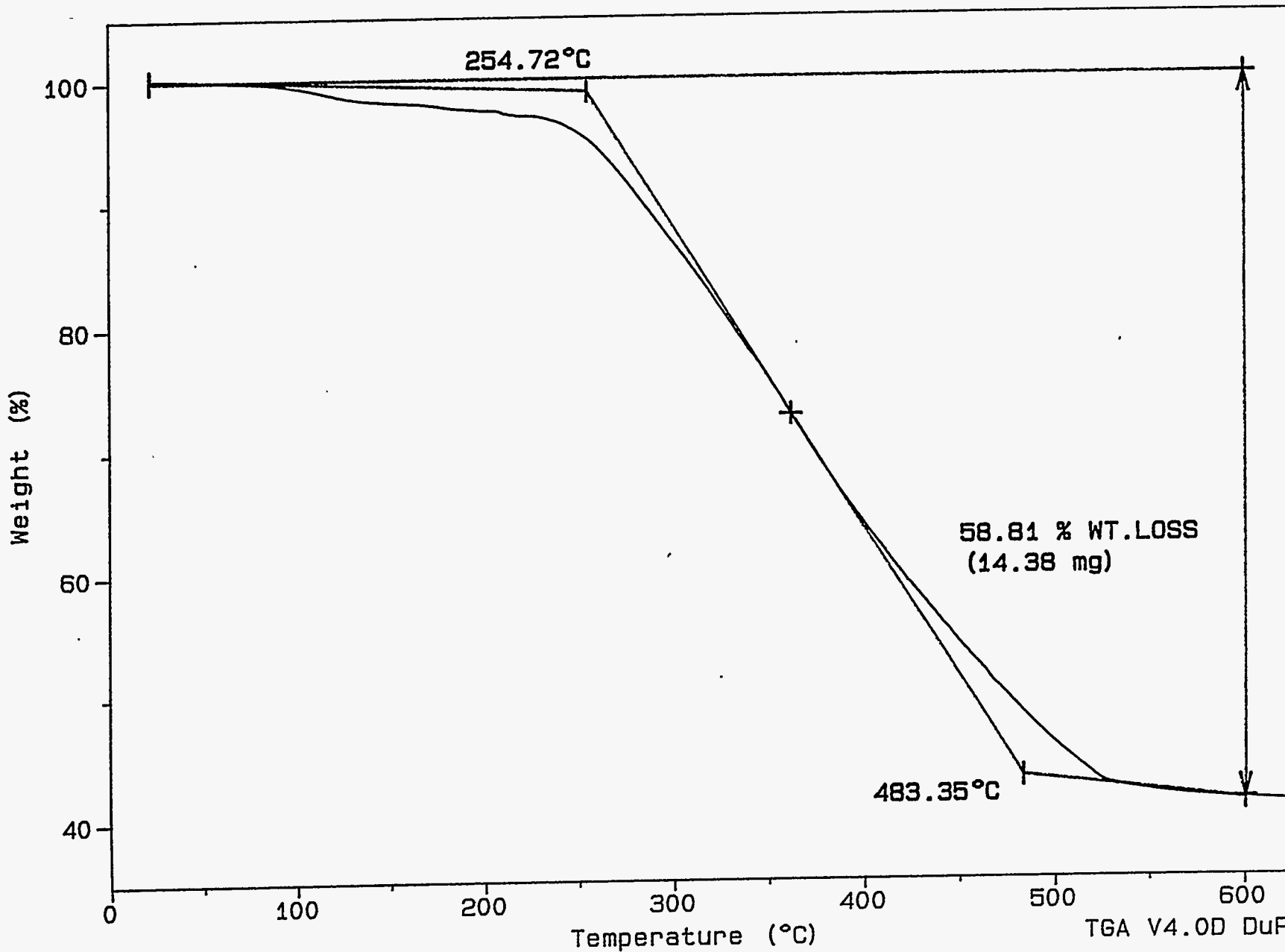
Operator: gfl

Method: 10C/MIN

Run Date: 23-Jun-95 08:34

Comment: ARGON 100CC/MIN Pt PAN

Figure 14: TGA Curve for C-300-6, Hydrotreated Coal  
450°C - 2 Hours, Fe<sub>2</sub>S<sub>3</sub> Catalyst



C300-9

Sample: 701-2-16-12

Size: 25.1950 mg

Method: 10C/MIN

Comment: ARGON 100CC/MIN Pt PAN

A95-01825 TGA

File: 95060801C.01

Operator: gfl

Run Date: 22-Jun-95 16:08

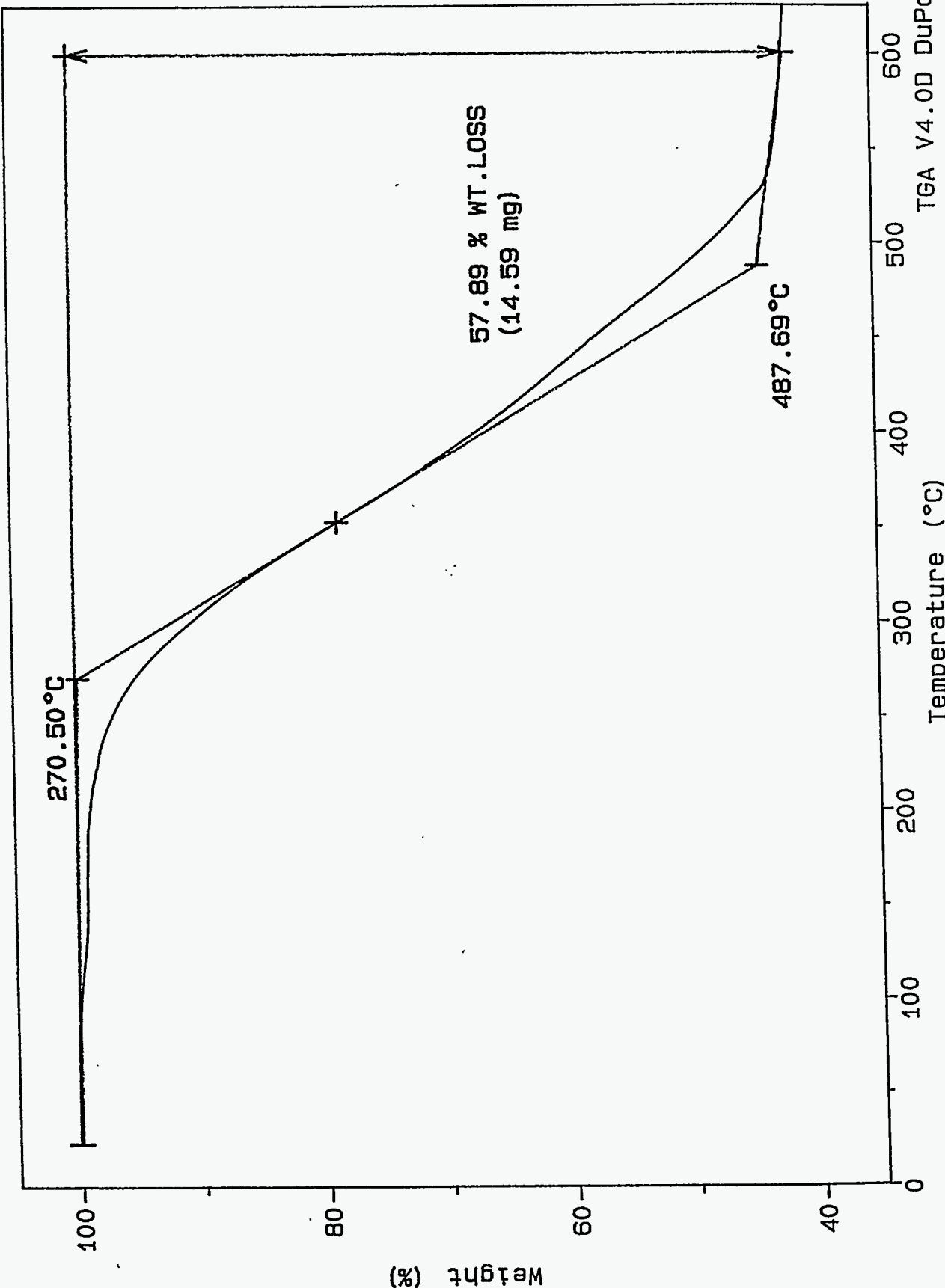
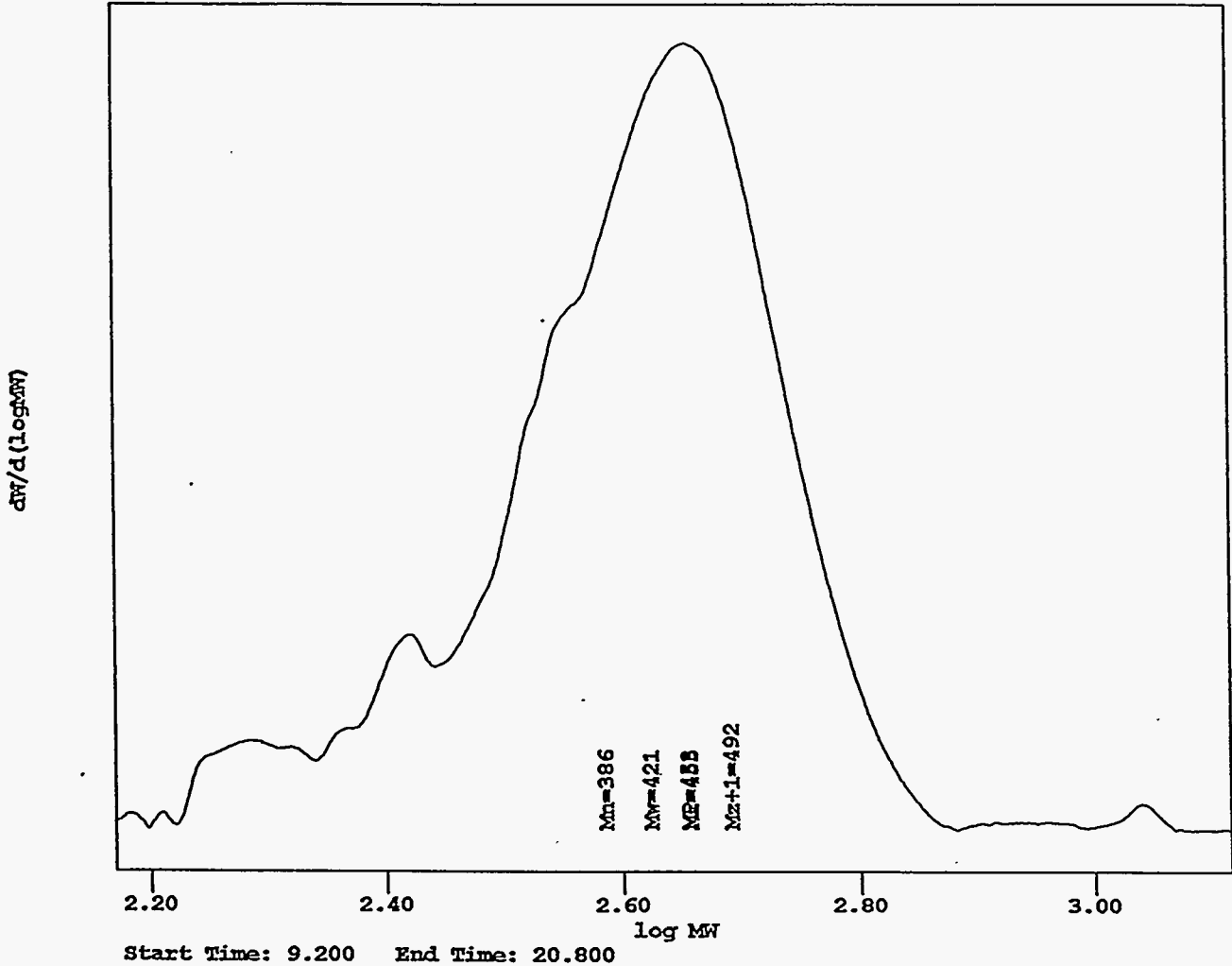


Figure 15: TGA Curve for C-300-9, Hydrotreated Coal  
450°C - 2 Hours, Mo(S) Catalyst



Project Name:	GPC	Sample Type:	Broad Unknown
Sample Name:	701-2-16-4	Volume:	100.00
Vial:	2	Run Time:	30.0 min
Injection:	1	Date Processed:	06/19/95 09:38:03 AM
Date Acquired:	06/16/95 12:43:30 PM	Pressure:	75
Processing Method:	GPC	Submitter:	RTL
Comments:	C300-3, Thermal 450-2 prepped 6/15/95 .25g/7ml TCB no fl. rt. mkr. 95060801		



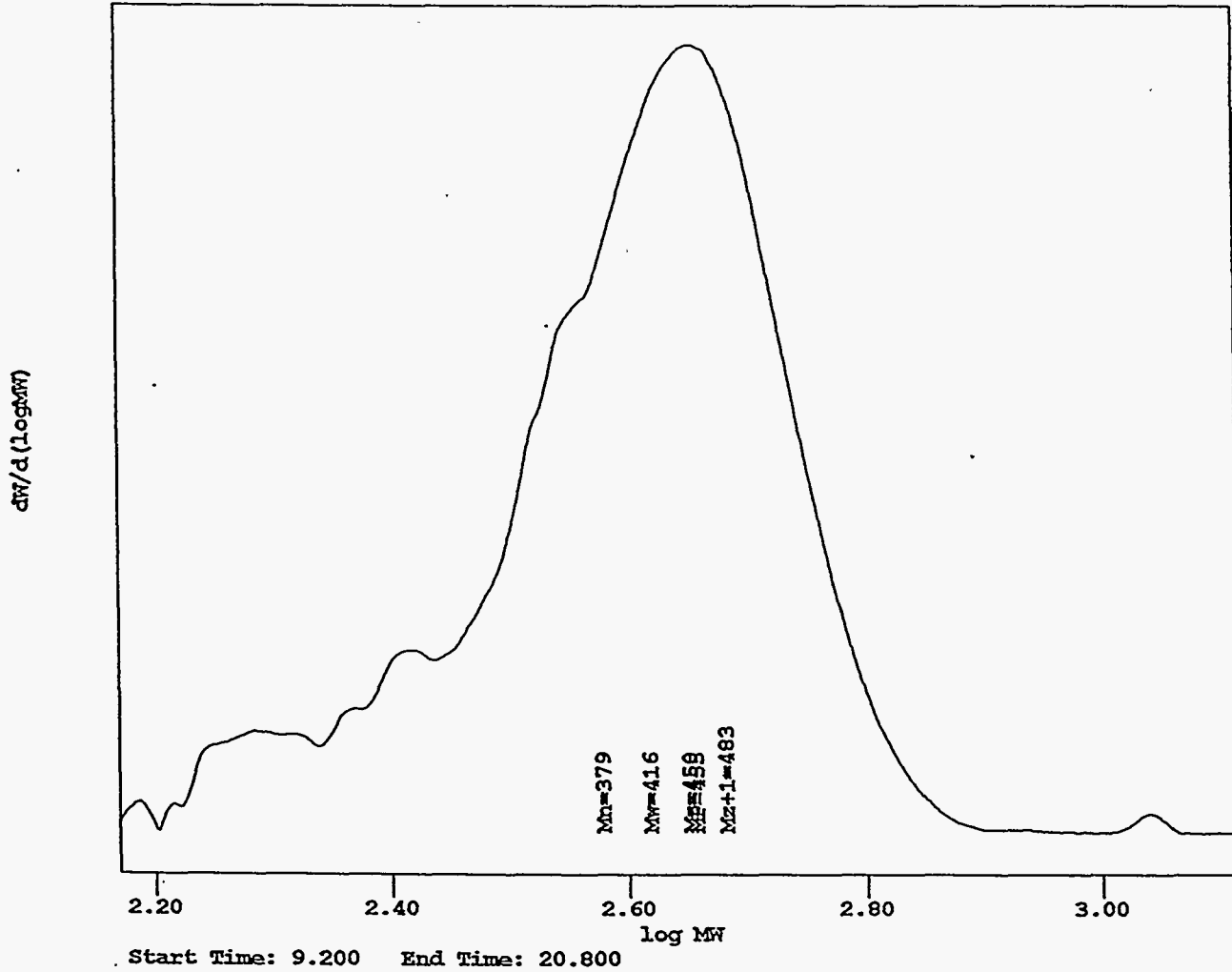
*Peak Results*

#	Ret Time (min)	Mn (Daltons)	MP (Daltons)	Mw (Daltons)	Mz (Daltons)	Mz+1 (Daltons)	Polydispersity
1	14.817	386	453	421	455	492	1.091709

Figure 16: Gel Permeation Chromatography Curve for C-300-3, Hydrotreated Coal 450°C - 2 Hours, No Catalyst

Project Name: GPC  
 Sample Name: 701-2-16-8  
 Vial: 3  
 Injection: 1  
 Date Acquired: 06/16/95 01:23:06 PM  
 Processing Method: GPC  
 Comments: C300-6, Fe Cat. 450-2 prepped 6/15/95 .25g/7ml TCB no fl. rt. mkr. 95060801

Sample Type: Broad Unknown  
 Volume: 100.00  
 Run Time: 30.0 min  
 Date Processed: 06/19/95 09:37:30 AM  
 Pressure: 75  
 Submitter: RTL



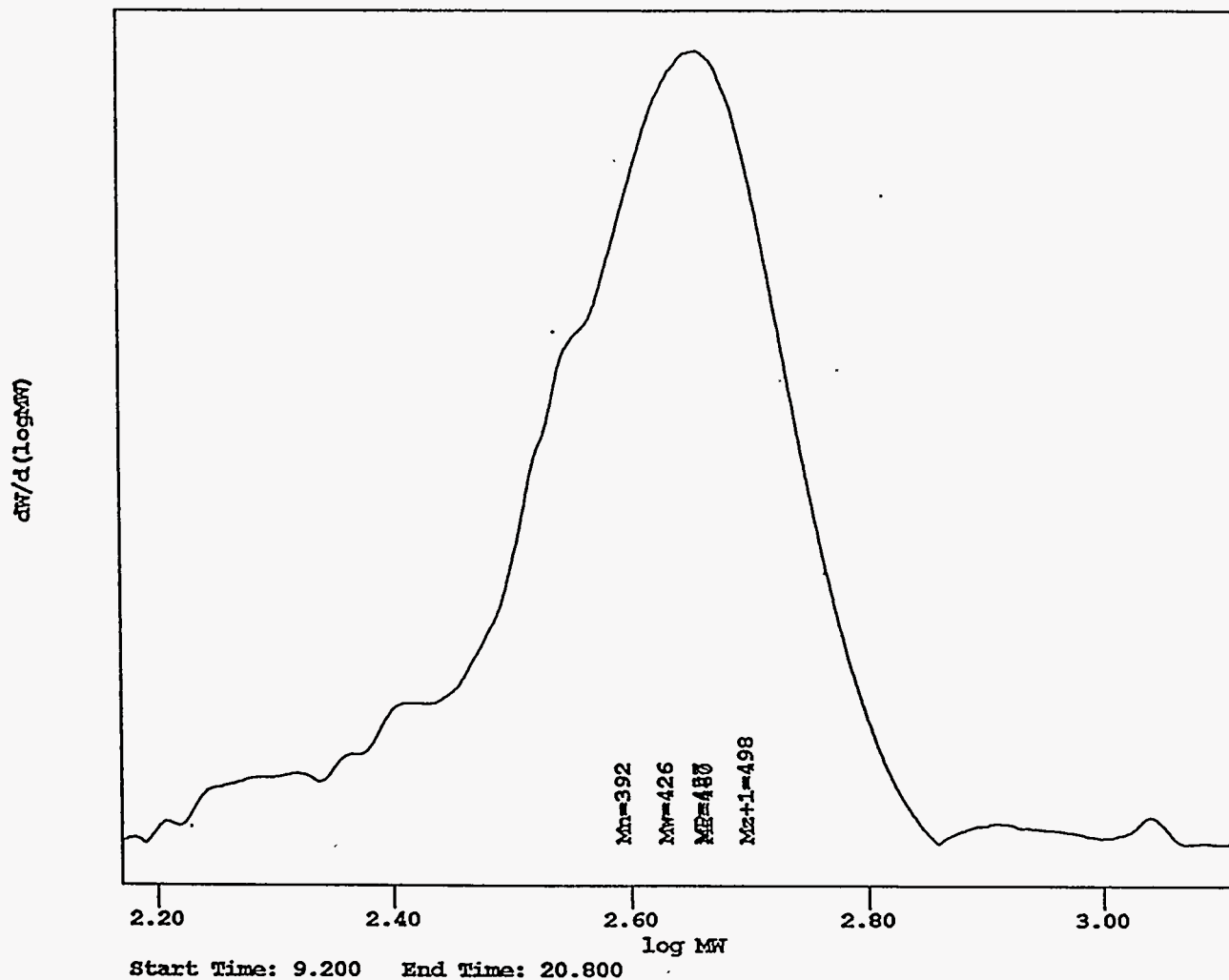
Peak Results

#	Ret Time (min)	Mn (Daltons)	MP (Daltons)	Mw (Daltons)	Mz (Daltons)	Mz+1 (Daltons)	Polydispersity
1	14.783	379	455	416	450	483	1.096545

Figure 17: Gel Permeation Chromatography Curve for C-300-6, Hydrotreated Coal 450°C - 2 Hours, Fe<sub>2</sub>S<sub>3</sub> Catalyst

Project Name: GPC  
 Sample Name: 701-2-16-12  
 Vial: 4  
 Injection: 1  
 Date Acquired: 06/16/95 01:55:19 PM  
 Processing Method: GPC  
 Comments: C300-9, Mo Cat. 450-2 prepped 6/15/95 .25g/7ml TCB no fl. rt. mkr. 95060801

Sample Type: Broad Unknown  
 Volume: 100.00  
 Run Time: 30.0 min  
 Date Processed: 06/19/95 09:36:59 AM  
 Pressure: 75  
 Submitter: RTL



Peak Results

#	Ret Time (min)	Mn (Daltons)	MP (Daltons)	Mw (Daltons)	Mz (Daltons)	Mz+1 (Daltons)	Polydispersity
1	14.767	392	457	426	460	498	1.087104

Figure 18: Gel Permeation Chromatography Curve for C-300-9, Hydrotreated Coal 450°C - 2 Hours, Mo(S) Catalyst

# PRODUCTS FROM 400°C-6 HR. HEAT TREATMENTS OF WVU COAL EXTRACTS

(Schematic Diagram)

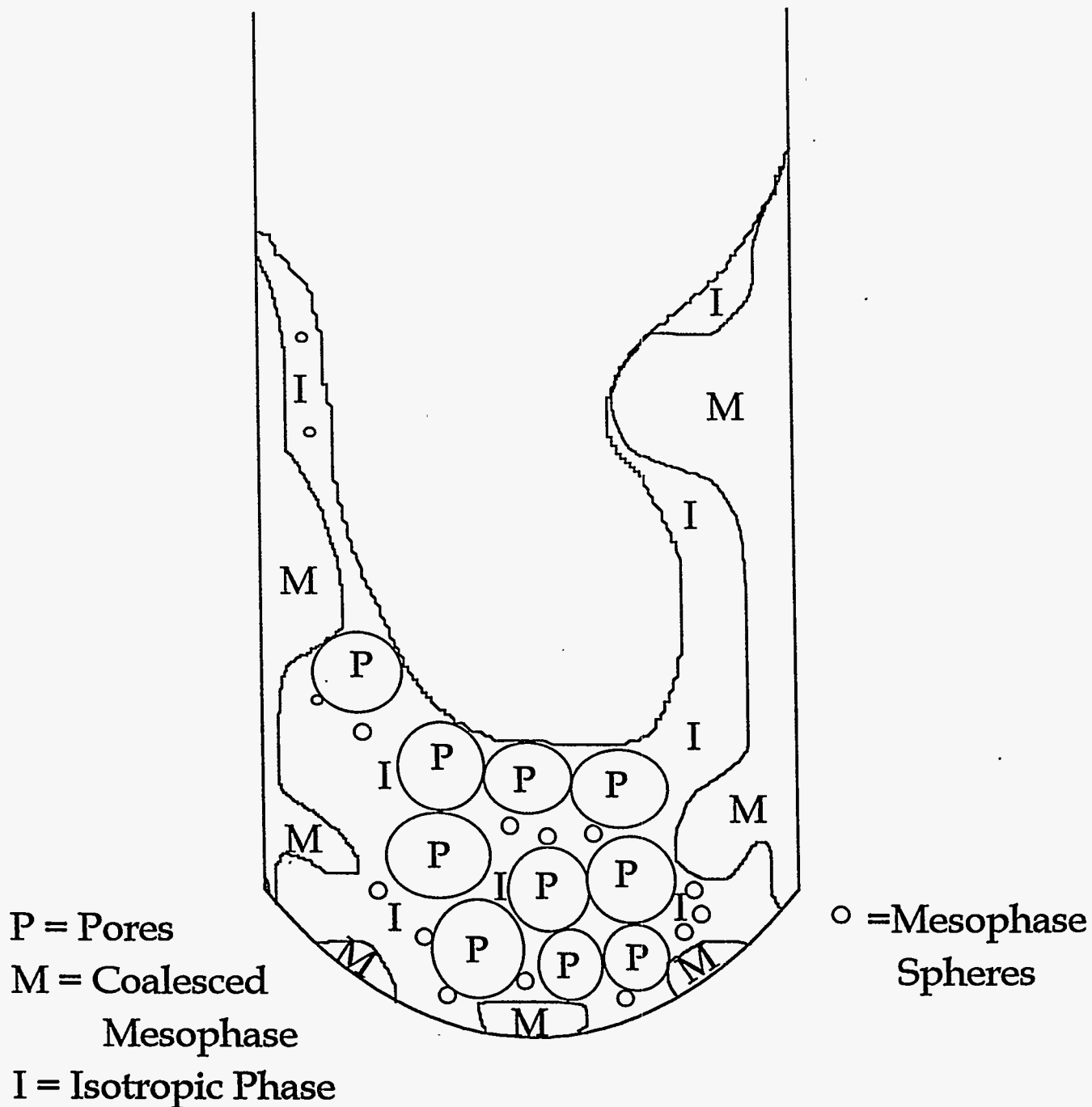
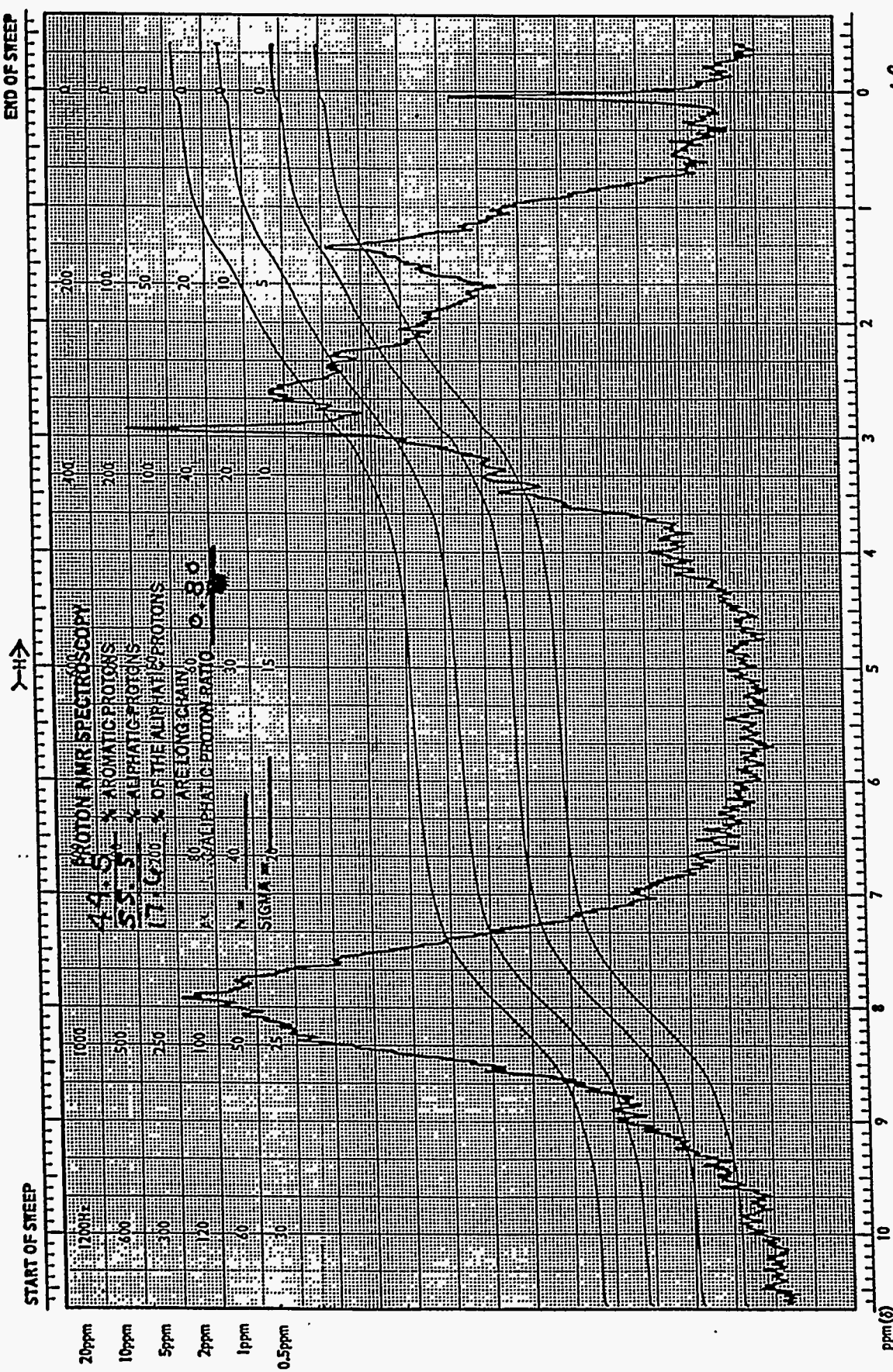


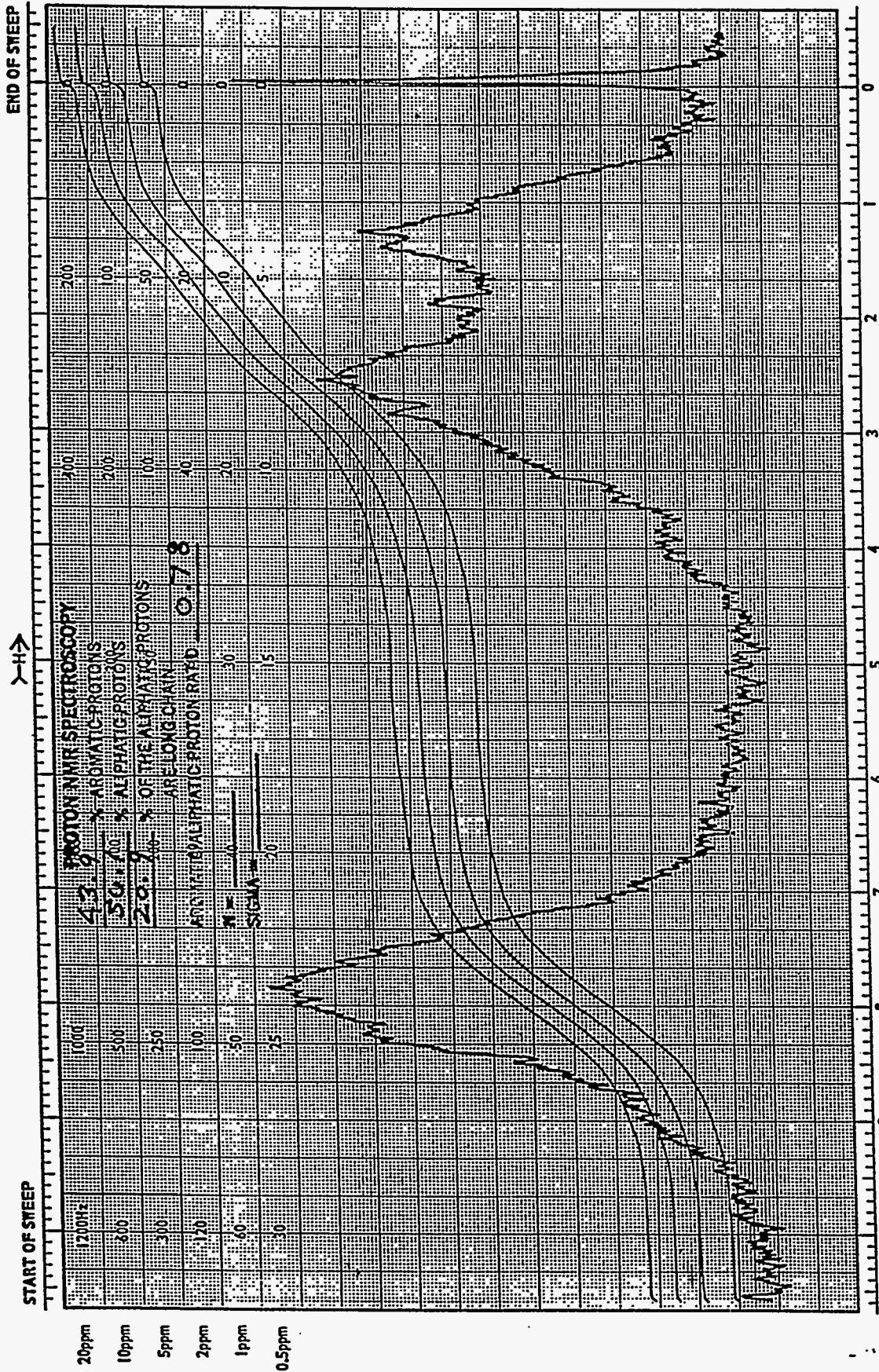
Figure 19



SPECTRUM AMPL. 2800 (90) SWEEP TIME 5 (1) min. SAMPLE: A076 (A95-03035) REMARKS: RLEWLS OPERATOR: gfl  
 FILTER 1 (OPEN) \_\_\_\_\_ sec SWEEP WIDTH 10 ppm or Hz SOLENT: CS2 DATE: 95/1/03  
 RF POWER 0.05 (0.1) mG END OF SWEEP 0 ppm or Hz DEC. LEVEL: TMS SPECTRUM NO. 95090209  
93% hot CS2  
SOLUBLE

WILMAD GLASS CO., INC.  
 U.S. Route 40 and Oak Road  
 BUREAU, N.J. 08310 U.S.A.  
 W-10-12/78  
 MADE IN U.S.A.  
 MCV-086K102

Figure 20: Proton NMR Spectrum for A076 from Hydrotreated Coal  
 WVG5-13421 - 450 °C, 2 Hours



OPERATOR gfl  
 DATE 95/103  
 SPECTRUM NO. 95090704

REMARKS: R. LEWIS  
 A066 (A95-03034)

93% hot CS<sub>2</sub>  
 soluble

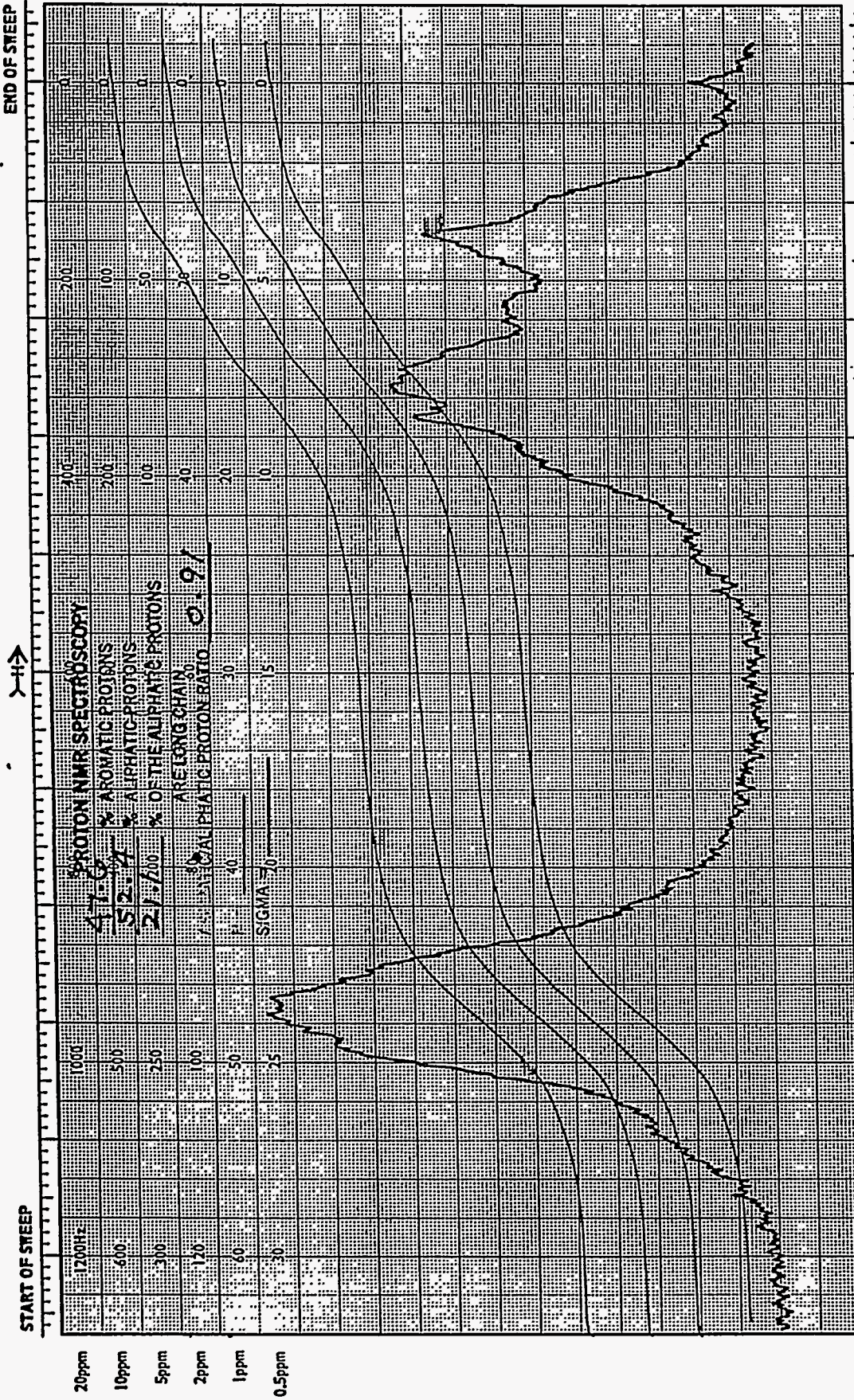
SAMPLE: CS<sub>2</sub>  
 TMS

SWEEP TIME 5 (1) min.  
 SWEEP WIDTH 10 ppm or Hz  
 END OF SWEEP 0 ppm or Hz

SPECTRUM AMPL. 2800 (90)  
 FILTER 1 (open) sec  
 RF POWER 0.05 (0.1) mG

WILMAD GLASS CO., INC.  
 U.S. FOUN. 40 and Oak Road  
 DUEEN, N.J. 08310 USA  
 PHILADELPHIA PA. 19104 USA  
 WCV40K132

Figure 21: Proton NMR Spectrum for A066 from Hydrotreated Coal  
 WVG5-13421 - 450°C, 1 Hour



START OF SWEEP

→

END OF SWEEP

20ppm  
10ppm  
5ppm  
2ppm  
1ppm  
0.5ppm

PROTON NMR SPECTROSCOPY  
 AROMATIC PROTONS  
 ALIPHATIC PROTONS  
 OF THE ALIPHATIC PROTONS  
 ARE LONG CHAIN  
 ALIPHATIC:PROTON RATIO 0.97

1700Hz  
600  
300  
120  
60  
30

1000  
500  
250  
100  
50  
25

400  
200  
100  
40  
20  
10  
5

200  
100  
50  
20  
10  
5

100  
50  
25  
10  
5

50  
25  
10  
5

25  
10  
5

10  
5

5

0

0

0

0

0

0

0

0

0

0

0

0

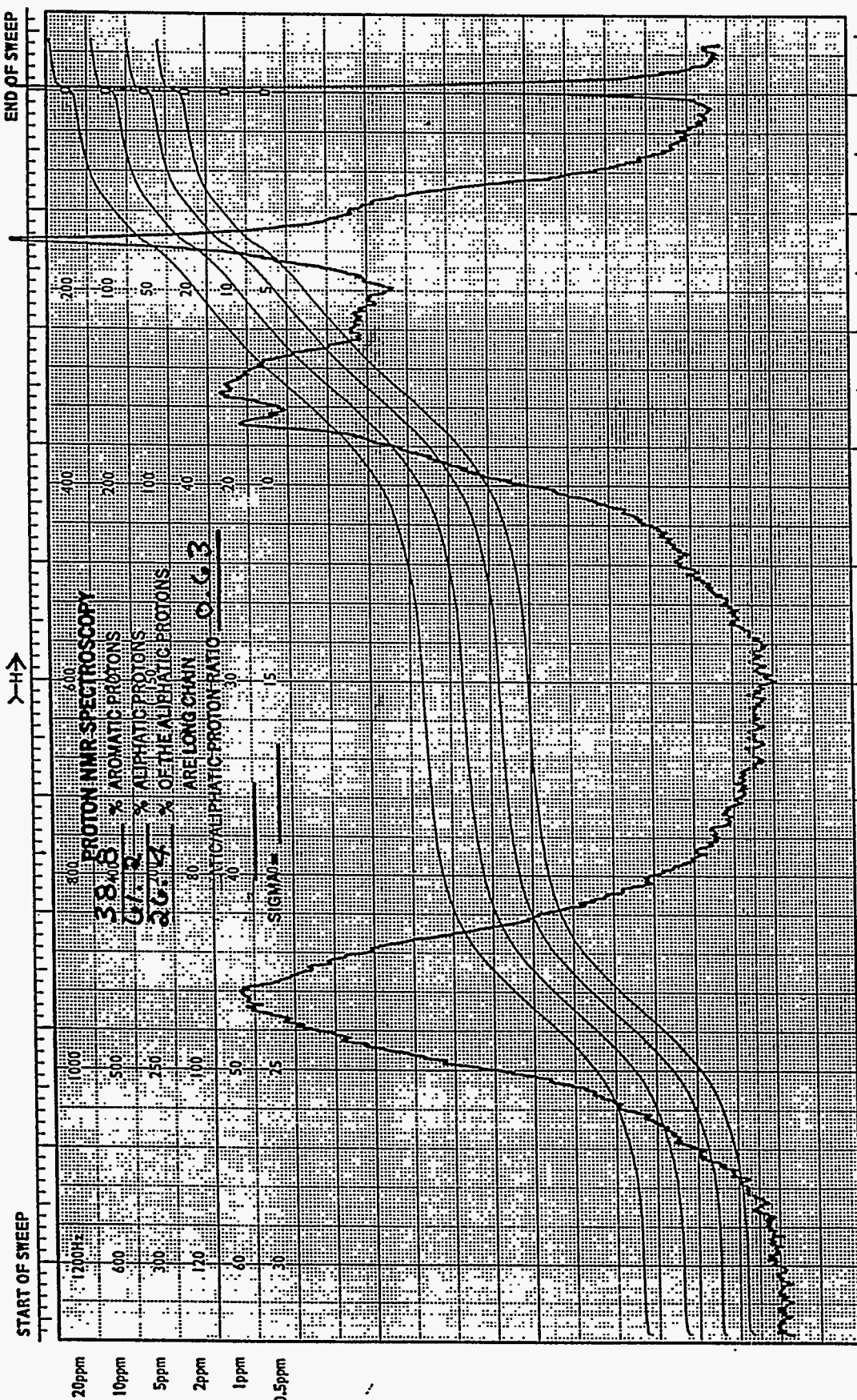
0

0

SPECTRUM AMPL. 1500 (50) SWEEP TIME 5 (1) min. SAMPLE: A073 (A95-03027) OPERATOR: RLF  
 FILTER 1 (OPEN) sec SWEEP WIDTH 10 ppm or Hz SOLVENT: CS2 TMS DATE 951102  
 RF POWER 0.05 (0.1) mG END OF SWEEP 0 ppm or Hz DEC. LEVEL: 94% hot CS2 SOLUBLE SPECTRUM NO. 95090104

WVAD GLASS CO. INC. U.S. FOUN. 40 AND Oak Road BUREAU H.I. 0810 U.S.A. W-16-1170

Figure 22: Proton NMR Spectrum for A073 from Hydrotreated Coal WVGS-13423 - 450°C, 2 Hours



20ppm  
10ppm  
5ppm  
2ppm  
1ppm  
0.5ppm

PROTON NMR SPECTROSCOPY  
 38.5% AROMATIC PROTONS  
 26.7% ALIPHATIC PROTONS  
 26.7% OF THE ALIPHATIC PROTONS  
 ARE LONG CHAIN  
 AROMATIC:ALIPHATIC PROTON RATIO 0.63

SIGMA = 15

PERKINELMER U.S.A. INC.  
 U.S. House 40 and Oak Road  
 BUENA VISTA, N.J. 08310 U.S.A.  
 WCV-360-K10-2

Figure 23: Proton NMR Spectrum for A075 from Hydrotreated Coal WUGS-13423 - 450°C, 1 Hour

START OF SWEEP →      ← END OF SWEEP

SPECTRUM AMPL. 1500 (50)      SWEEP TIME 5 (1) min.      SAMPLE: A075 (A95-03036)      REMARKS: R. LEWIS  
 FILTER 1 (OPEN) sec      SWEEP WIDTH 10 ppm or Hz      SOLVENT: CS2      TMS      70% hot CS2 soluble  
 RF POWER 0.05 (0.1) mG      END OF SWEEP 0 ppm or Hz      DEC. LEVEL

OPERATOR off      DATE 951102      SPECTRUM NO. 95090704



Sample: A076 701-2-30-2

A95-03035 TGA

File: 95090704B.01

Size: 18.5080 mg

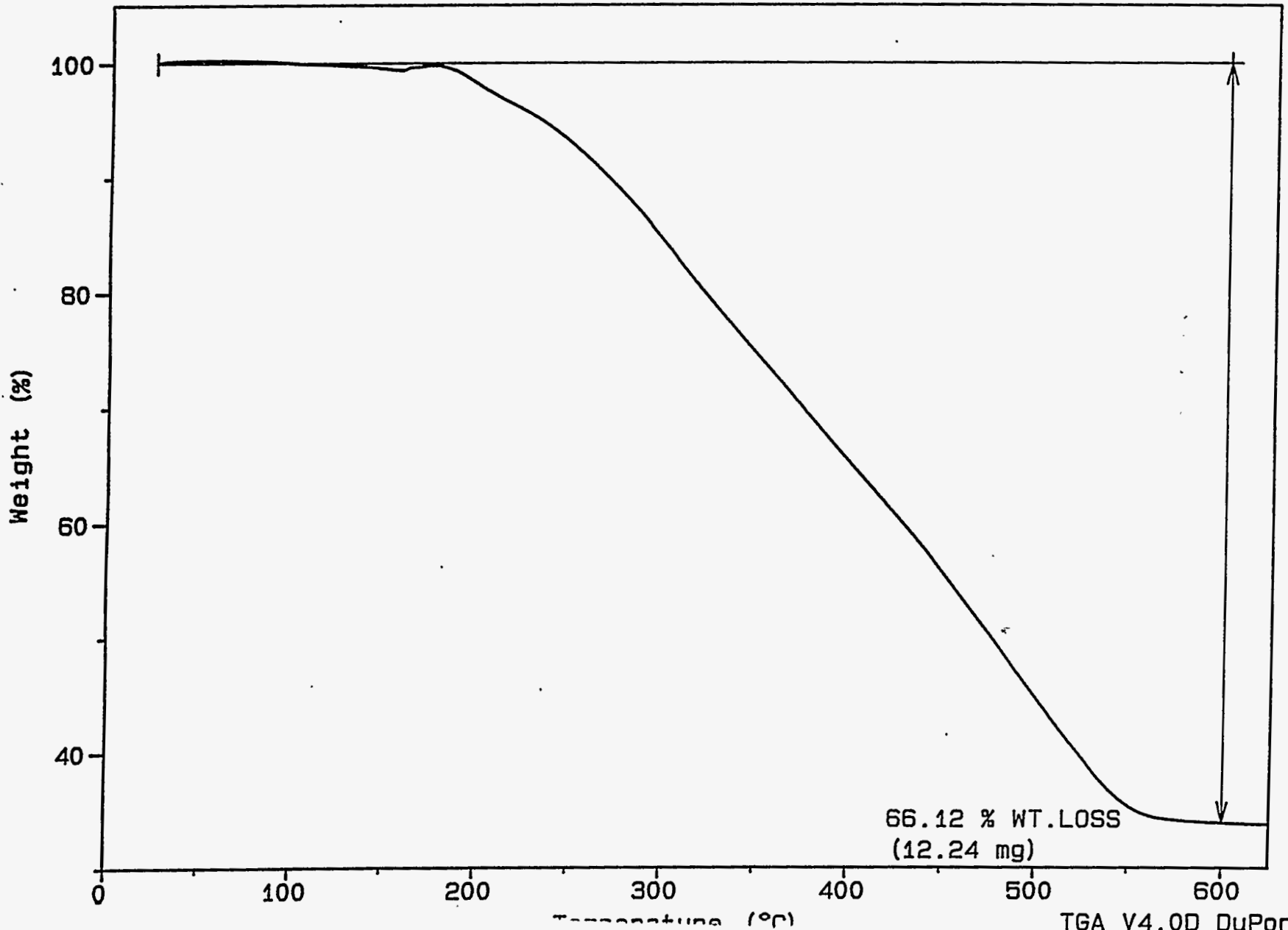
Operator: gfl

Method: 10°C/MIN

Run Date: 16-Oct-95 11:52

Comment: ARGON 100CC/MIN Pt PAN

Figure 24: TGA Curve for A076 from Hydrotreated Coal  
WVGS-13421 - 450°C, 2 Hours



Sample: A066 701-2-30-1

A95-03034 TGA

File: 95090704A.02

Size: 24.6620 mg

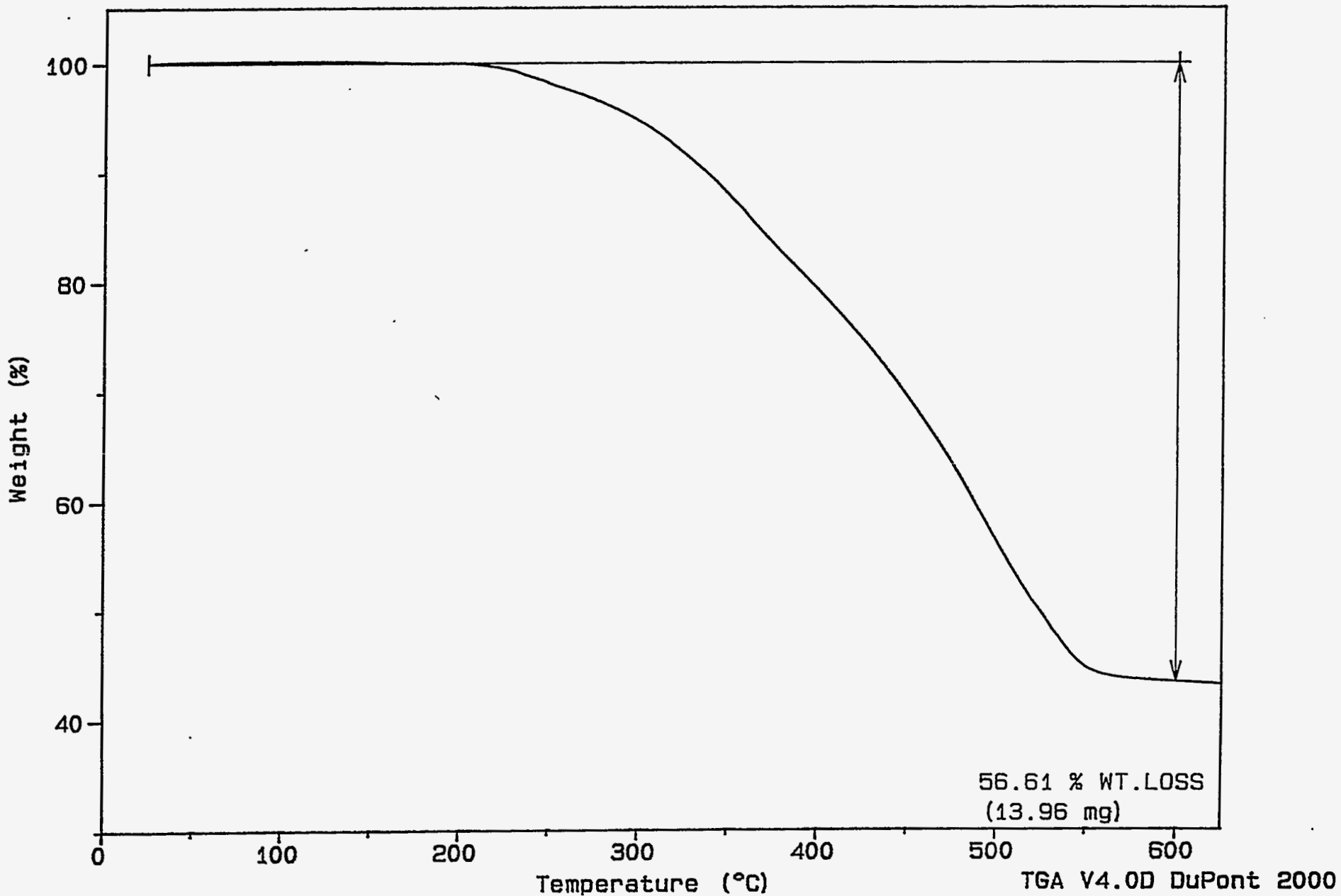
Operator: gfl

Method: 10°C/MIN

Run Date: 16-Oct-95 09:35

Comment: ARGON 100CC/MIN Pt PAN

Figure 25: TGA Curve for A066 from Hydrotreated Coal  
WVGS-13421 - 450°C, 1 Hour



Sample: A073 701-2-30-3

Size: 19.3970 mg

Method: 10°C/MIN

Comment: ARGON 100CC/MIN Pt PAN

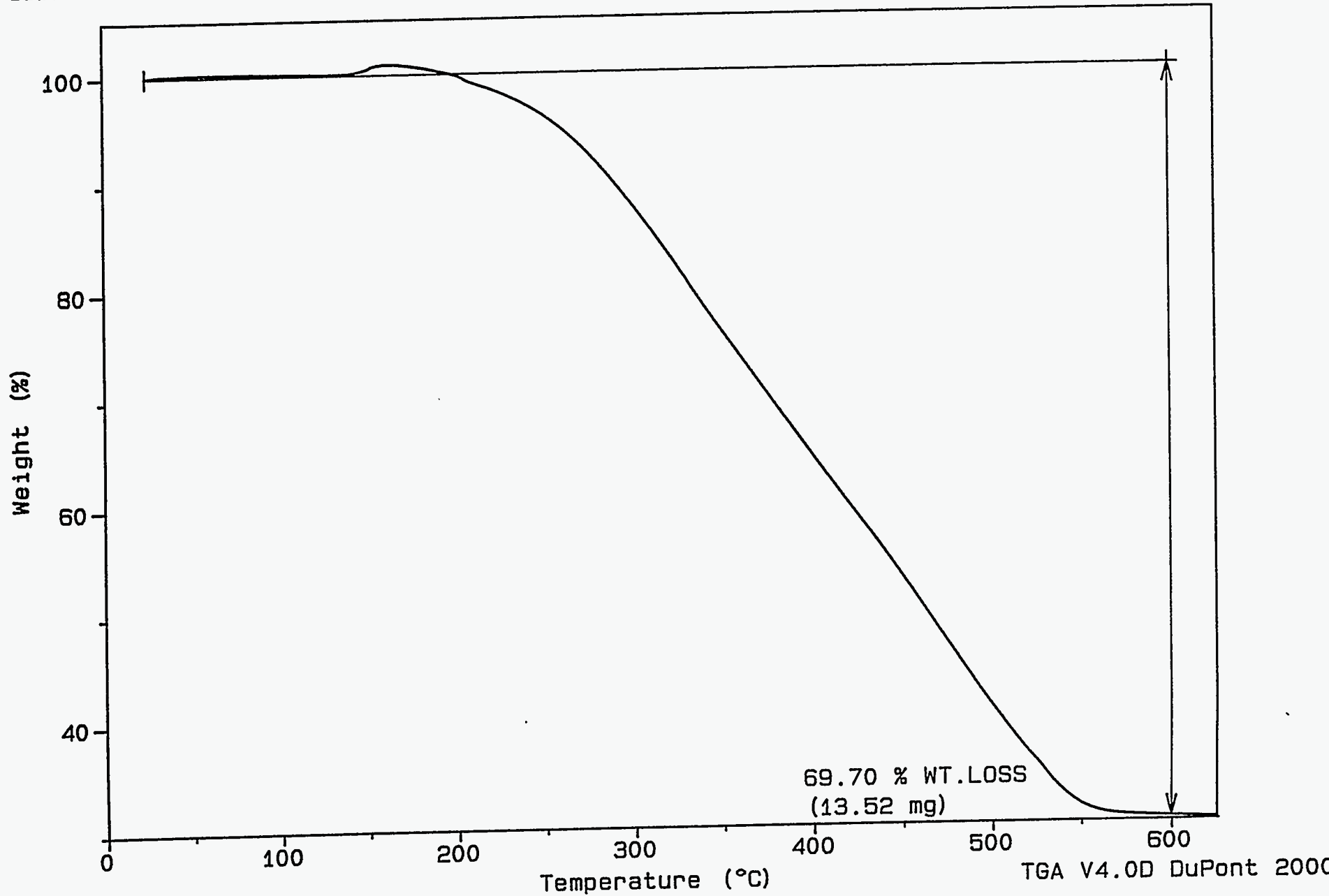
A95-03037 TGA

File: 95090704D.02

Operator: gfl

Run Date: 18-Oct-95 08:49

Figure 26: TGA Curve for A073 from Hydrotreated Coal  
MWGS-13423 - 450°C, 2 Hours



Sample: A075 701-2-30-3

A95-03036 TGA

File: 95090704C.02

Size: 19.0000 mg

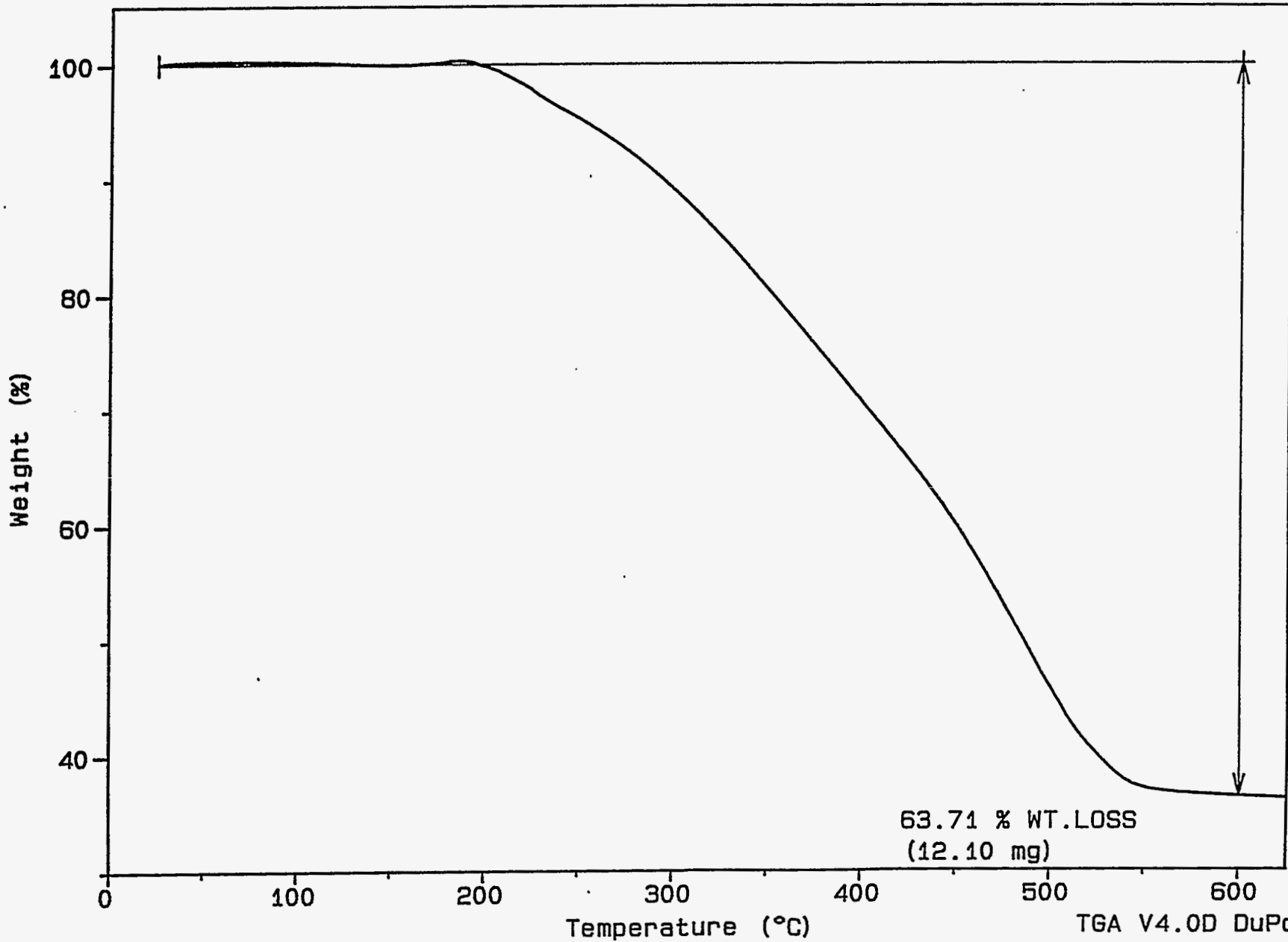
Operator: gfl

Method: 10°C/MIN

Run Date: 17-Oct-95 16:52

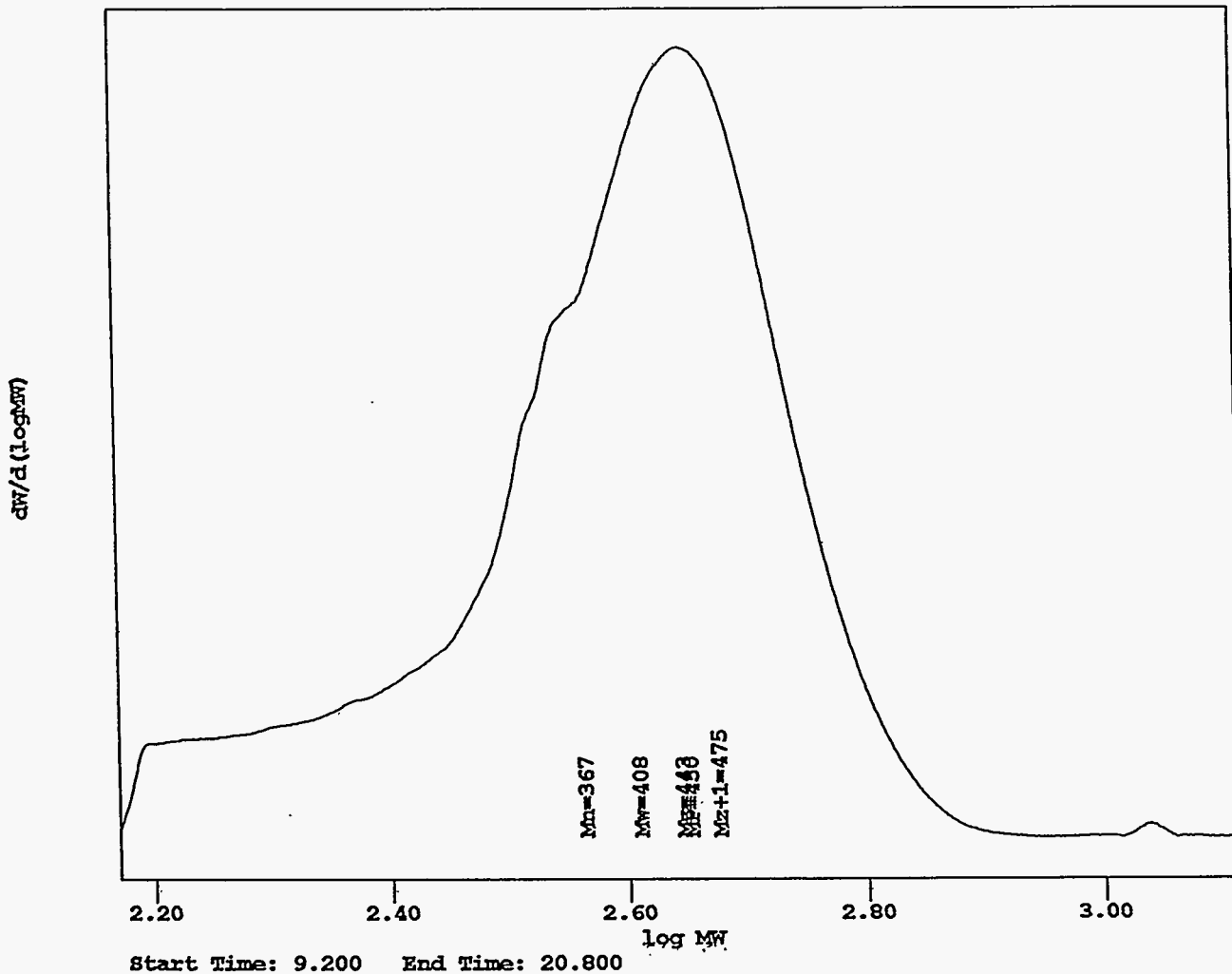
Comment: ARGON 100CC/MIN Pt PAN

Figure 27: TGA Curve for A075 from Hydrotreated Coal  
WVGS-13423 - 450°C, 1 Hour



Project Name: GPC  
 Sample Name: A076  
 Vial: 4  
 Injection: 1  
 Date Acquired: 09/25/95 02:08:13 PM  
 Processing Method: GPC  
 Comments: 701-2-30-2 (WVU) prepped 7/21/95

Sample Type: Broad Unknown  
 Volume: 100.00  
 Run Time: 30.0 min  
 Date Processed: 09/25/95 03:49:51 PM  
 Pressure: 73  
 Submitter: rtl



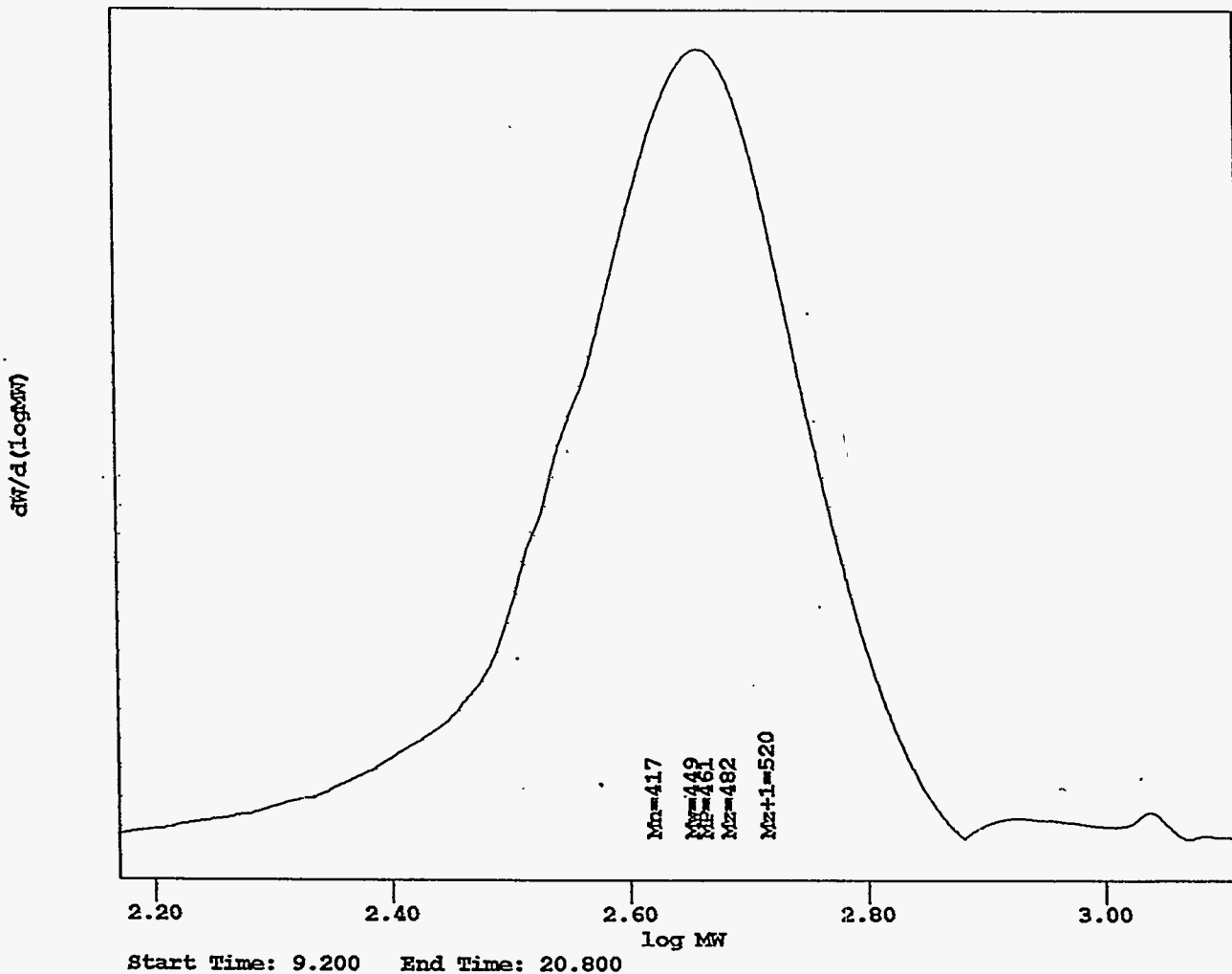
*Peak Results*

#	Ret Time (min)	Mn (Daltons)	MP (Daltons)	Mw (Daltons)	Mz (Daltons)	Mz+1 (Daltons)	Polydispersity
1	14.850	367	450	408	443	475	1.111614

Figure 28: Gel Permeation Chromatogram for A076 from Hydrotreated Coal WVGS-13421 - 450°C, 2 Hours

Project Name: GPC  
 Sample Name: A066  
 Vial: 3  
 Injection: 1  
 Date Acquired: 09/25/95 01:11:58 PM  
 Processing Method: GPC  
 Comments: 701-2-30-1(WVU) prepped 7/21/95

Sample Type: Broad Unknown  
 Volume: 100.00  
 Run Time: 30.0 min  
 Date Processed: 09/25/95 03:48:09 PM  
 Pressure: 73  
 Submitter: rtl



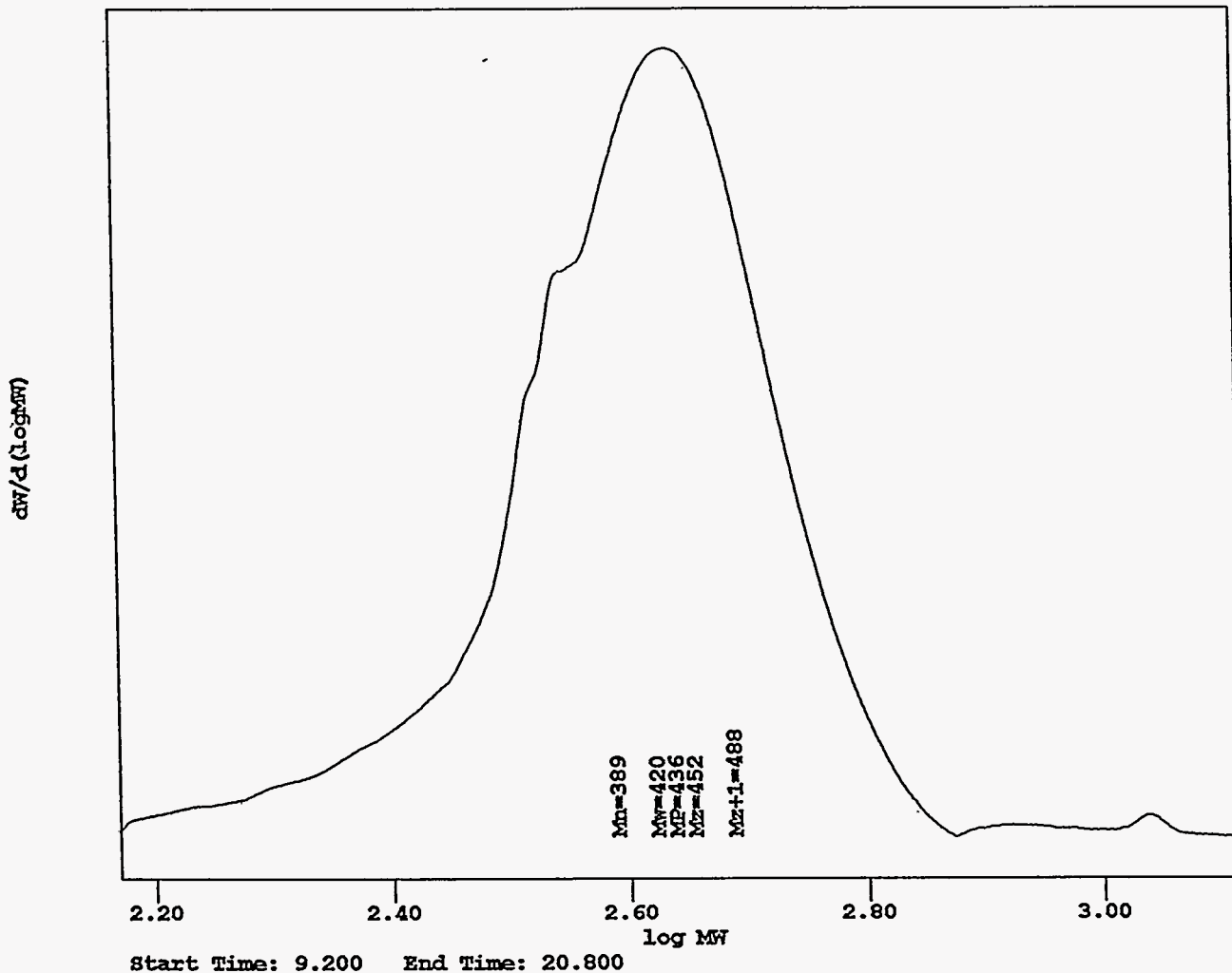
*Peak Results*

#	Ret Time (min)	Mn (Daltons)	MP (Daltons)	Mw (Daltons)	Mz (Daltons)	Mz+1 (Daltons)	Polydispersity
1	14.717	417	461	449	482	520	1.076375

Figure 29: Gel Permeation Chromatogram for A066 from Hydrotreated Coal WVGS-13421 - 450°C, 1 Hour

Project Name: GPC  
 Sample Name: A073  
 Vial: 6  
 Injection: 1  
 Date Acquired: 09/25/95 03:15:40 PM  
 Processing Method: GPC  
 Comments: 701-2-30-4 (WVU) prepped 7/21/95

Sample Type: Broad Unknown  
 Volume: 100.00  
 Run Time: 30.0 min  
 Date Processed: 09/25/95 03:48:35 PM  
 Pressure: 73  
 Submitter: rtl



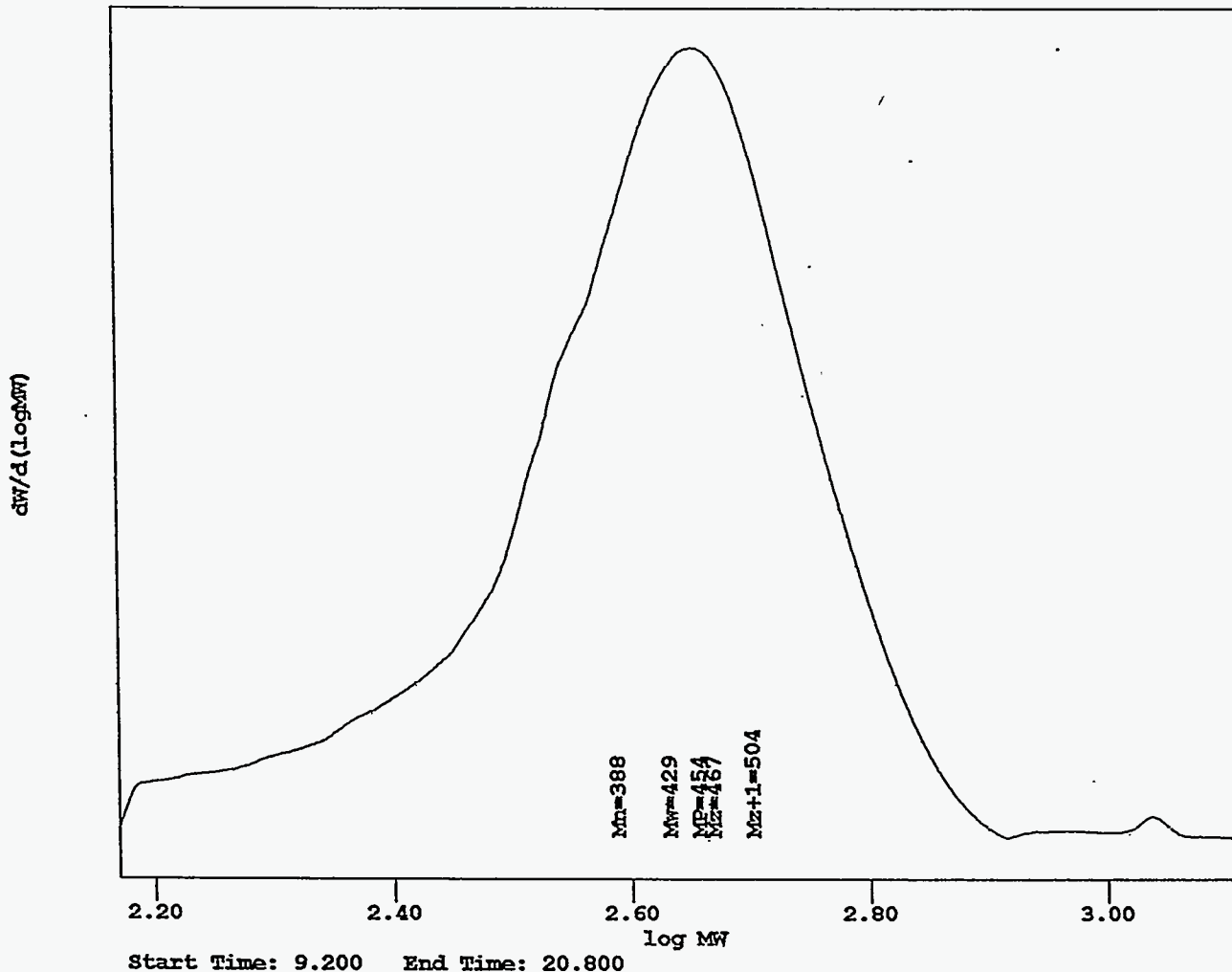
Peak Results

#	Ret Time (min)	Mn (Daltons)	MP (Daltons)	Mw (Daltons)	Mz (Daltons)	Mz+1 (Daltons)	Polydispersity
1	15.017	389	436	420	452	488	1.079811

Figure 30: Gel Permeation Chromatogram for A073 from Hydrotreated Coal WVGS-13423 - 450°C, 2 Hours

Project Name: GPC  
 Sample Name: A075  
 Vial: 5  
 Injection: 1  
 Date Acquired: 09/25/95 02:44:30 PM  
 Processing Method: GPC  
 Comments: 701-2-30-3 (WVU) prepped 7/21/95

Sample Type: Broad Unknown  
 Volume: 100.00  
 Run Time: 30.0 min  
 Date Processed: 09/25/95 03:49:10 PM  
 Pressure: 73  
 Submitter: rtl

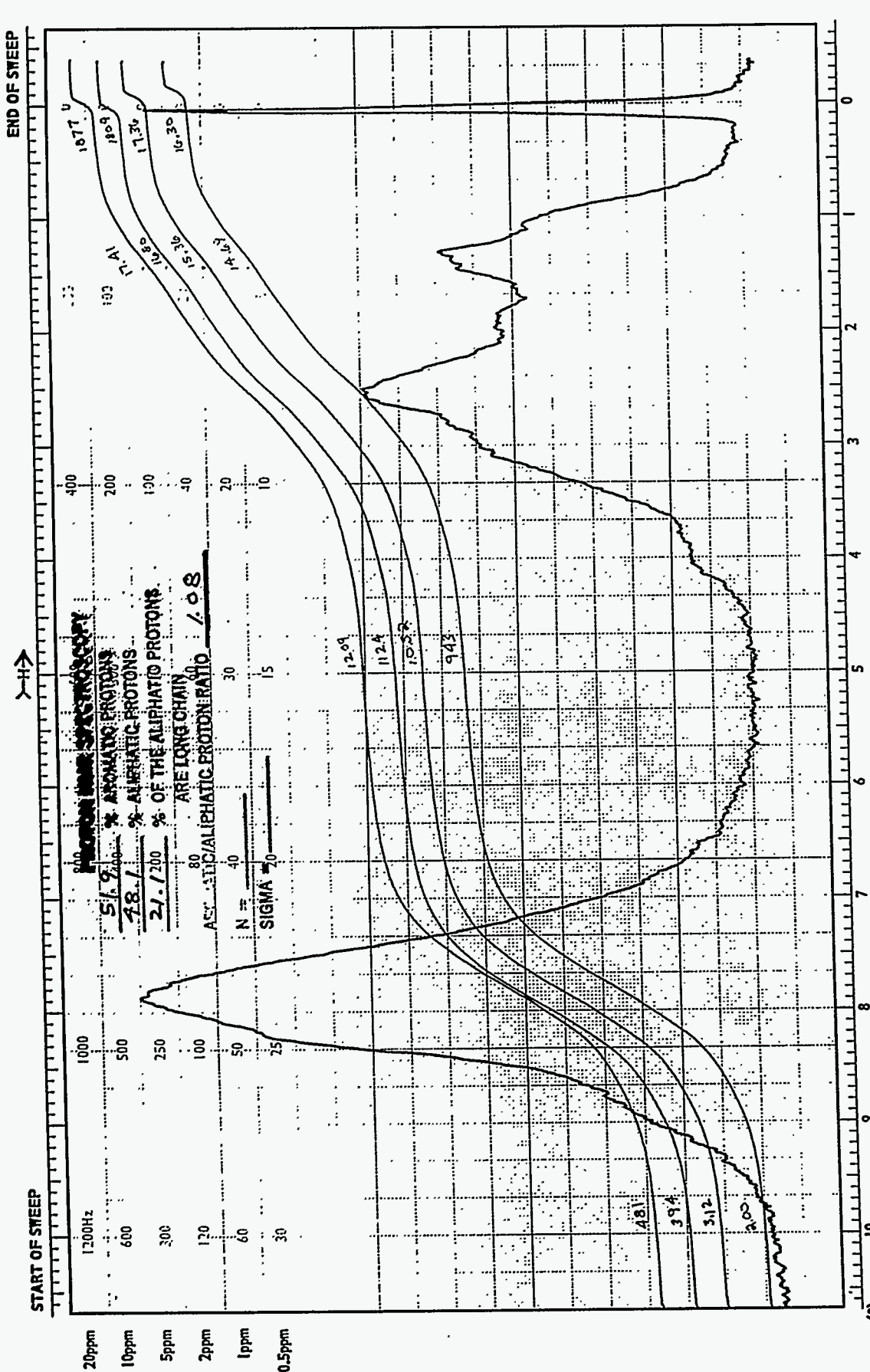


*Peak Results*

#	Ret Time (min)	Mn (Daltons)	MP (Daltons)	Mw (Daltons)	Mz (Daltons)	Mz+1 (Daltons)	Polydispersity
1	14.800	388	454	429	467	504	1.106079

Figure 31: Gel Permeation Chromatogram for A075 from Hydrotreated Coal WVGS-13423 - 450°C, 1 Hour

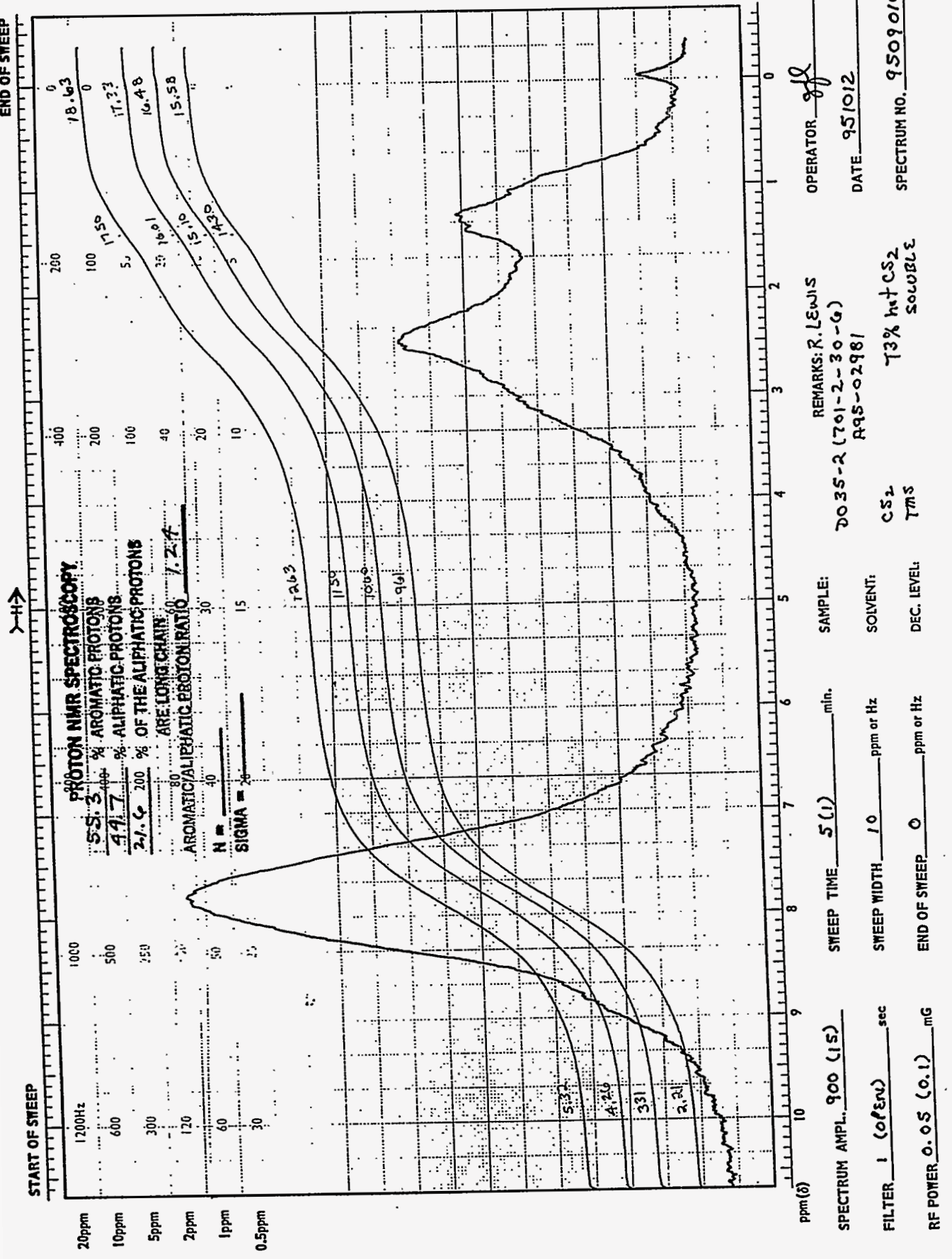




OPERATOR JLL  
 DATE 9/5/012  
 SPECTRUM NO. 95090101  
 REMARKS: R.L. Lewis  
 SAMPLE: D035-1 (701-2-30-5)  
R95-02980  
 SOLVENT: CS2  
TMS  
93% hot CS2  
SOLUBLE  
 SWEPT AMPL. 900 (15) sec  
 FILTER 1 (OPEN) ppm or Hz  
 SWEEP TIME 5 (1) min.  
 SWEEP WIDTH 10 ppm or Hz  
 END OF SWEEP 0 ppm or Hz  
 RF POWER 0.05 (0.1) mG  
 DEC. LEVEL:

WILMAD GLASS CO., INC.  
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 BUENA, N.J. 08310 U.S.A.  
 WILMAD U.S.A.  
 WCV-360-K10-2  
 M-100-12/77

Figure 32: Proton NMR Spectrum for 250°C Distillation Residue D035-1



START OF SWEEP →

← END OF SWEEP

OPERATOR off

DATE 951012

SPECTRUM NO. 95090101

REMARKS: R. LEWIS

D035-2 (701-2-30-G)

R95-02981

73% mt CS<sub>2</sub>

CS<sub>2</sub>

TMS

SAMPLE: 5 (1) min.

SOLVENT: 10 ppm or Hz

DEC. LEVEL: 0 ppm or Hz

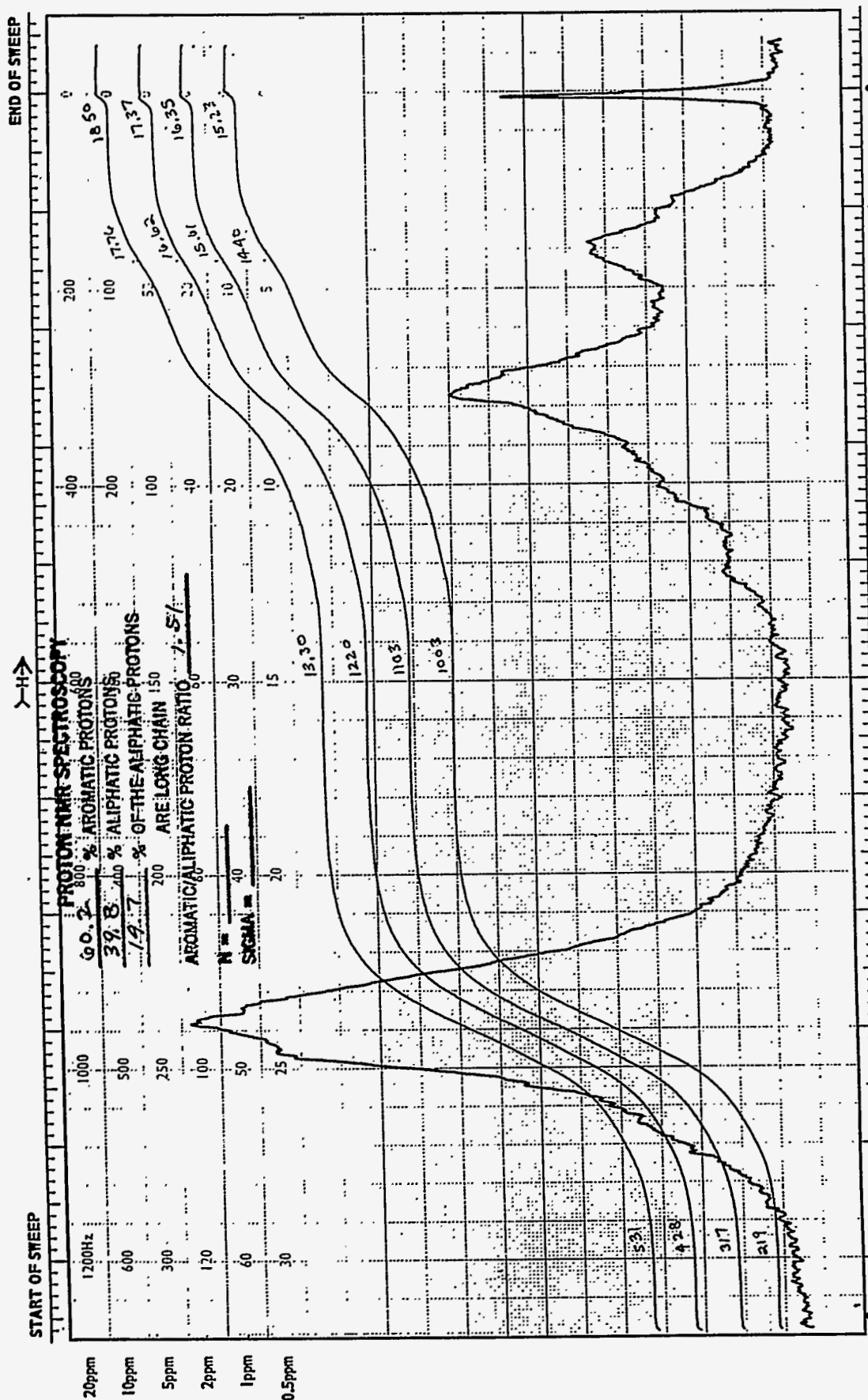
SPECTRUM AMPL. 900 (15)

FILTER 1 (0.5V) sec

RF POWER 0.05 (0.1) mG

WILMAD GLASS CO., INC.  
 U.S. Route 40 and Oak Road  
 BUENA, N.J. 08310 U.S.A.  
 WILMAD  
 W-100-1270

Figure 33: Proton NMR Spectrum for 350°C Distillation Residue D035-2



WILMAD GLASS CO., INC.  
 U.S. Route 40 and Oak Road  
 BUENA, N.J. 08310 U.S.A.  
 Printed in U.S.A.  
 WCV-360-K10-2  
 M-10-12/73

OPERATOR R. L. LEWIS  
 DATE 9/5/11  
 SPECTRUM NO. 95090101

REMARKS: D035-3 (701-2-30-7)  
R95-02982  
6.5% hot CS<sub>2</sub>  
SOLUBLE

SAMPLE: D035-3 (701-2-30-7)  
 SOLVENT: CS<sub>2</sub>  
 DEC. LEVEL: TMS  
 SPECTRUM AMPL. 1000 (2.5) min.  
 SWEEP TIME 5 (1) ppm or Hz  
 FILTER 1 (OPEN) sec  
 SWEEP WIDTH 10 ppm or Hz  
 RF POWER 0.05 (0.1) mg  
 END OF SWEEP 0 ppm or Hz

Figure 34: Proton NMR Spectrum for 400°C Distillation Residue D035-3

Sample: D035-1 (701-2-30-5)

A95-02980

TGA

File: A: 95090101A.02

Size: 26.1319 mg

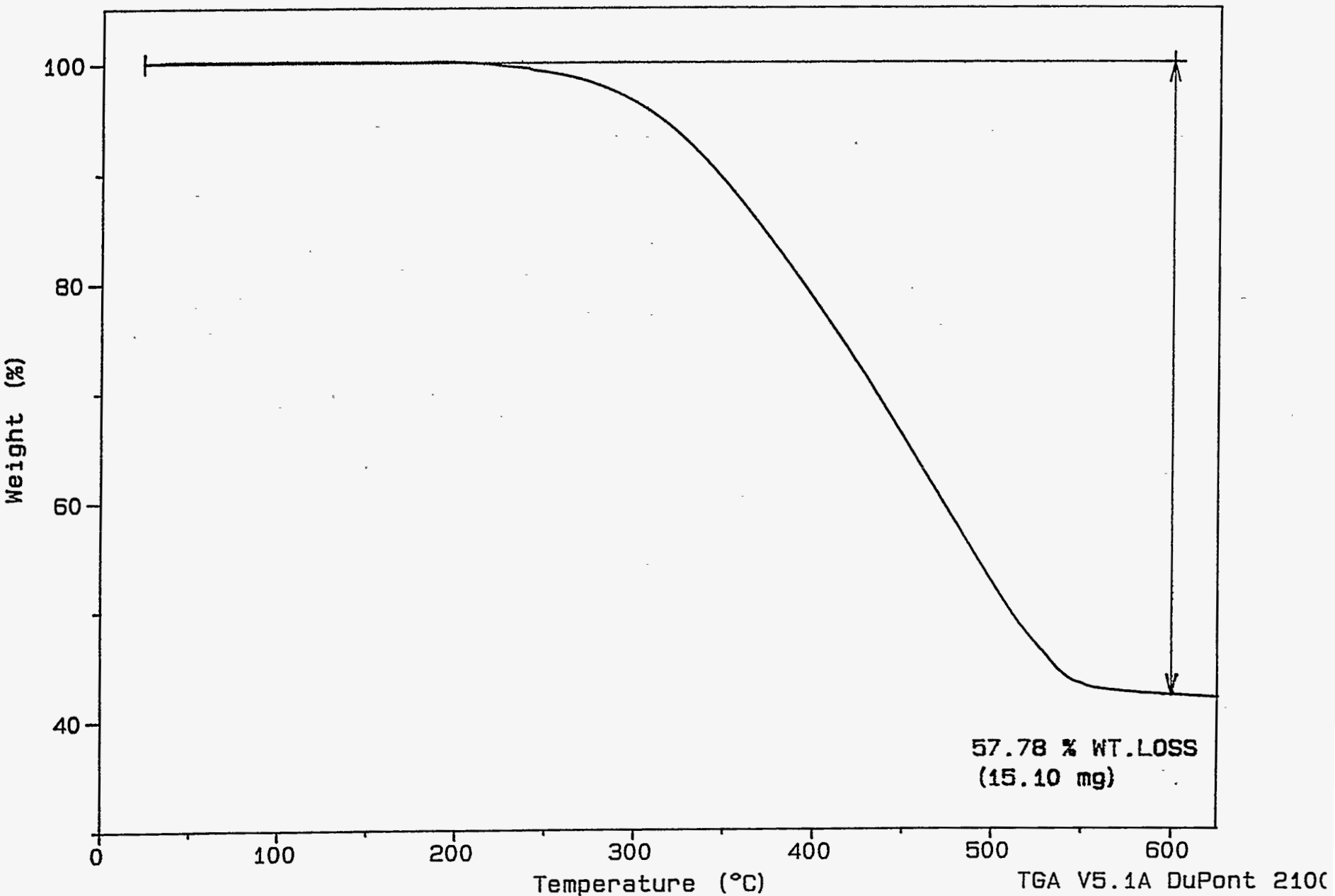
Operator: gfl

Method: 10°C/MIN

Run Date: 6-Sep-95 11:48

Comment: ARGON 100CC/MIN Pt PAN

Figure 35: TGA Curve for 250°C Distillation Residue D035-1



Sample: D035-2 (701-2-30-6)

A95-02981 TGA

File: A:95090101B.02

Size: 26.2477 mg

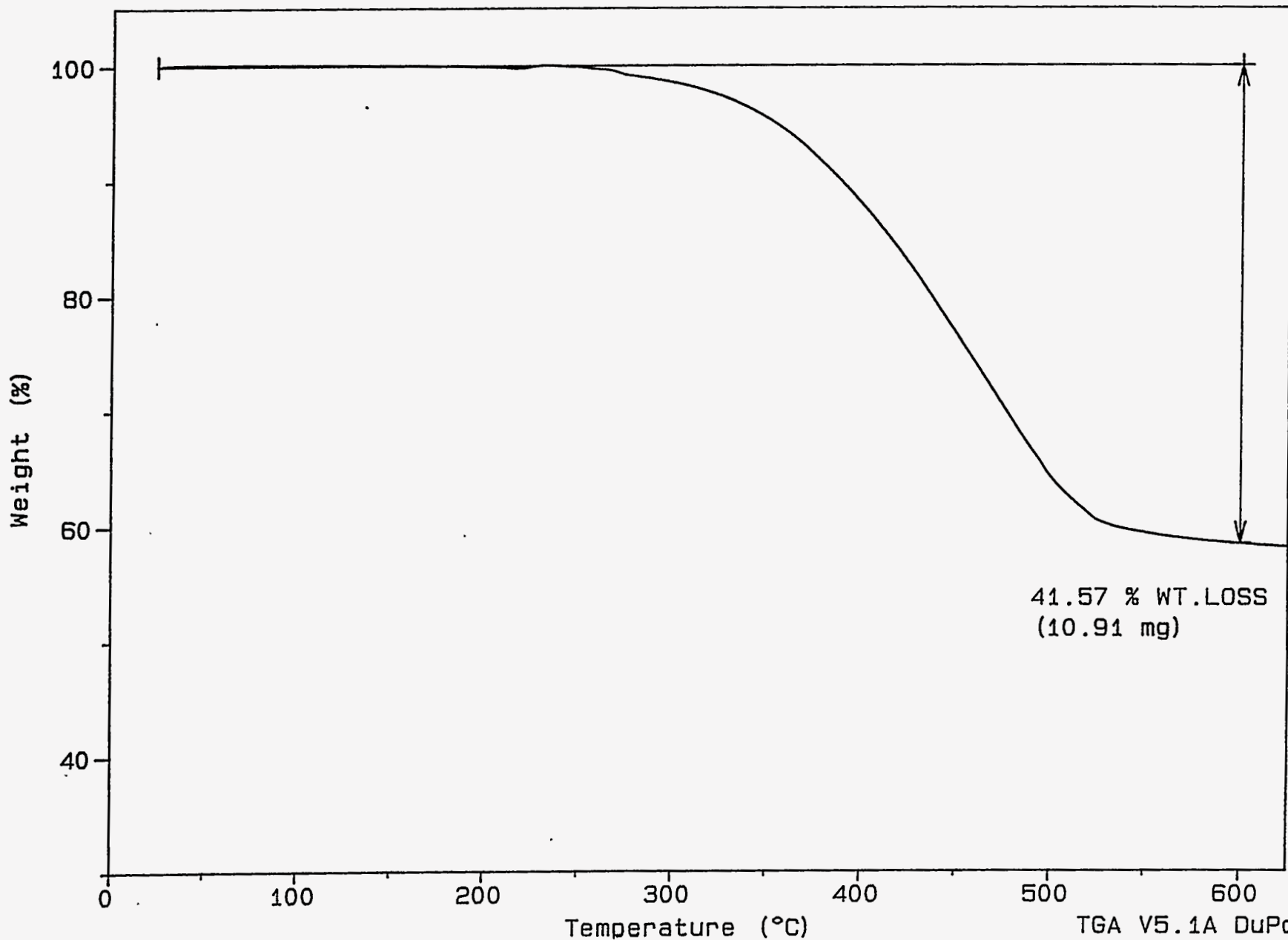
Operator: gfl

Method: 10 °C/MIN

Run Date: 6-Sep-95 14:12

Comment: ARGON 100CC/MIN Pt PAN

Figure 36: TGA Curve for 350°C Distillation Residue D035-2



41.57 % WT. LOSS  
(10.91 mg)

Sample: D035-3 (701-2-30-7)

A95-02982 TGA

File: A: 95090101C.01

Size: 25.0206 mg

Operator: gfl

Method: 10°C/MIN

Run Date: 5-Sep-95 12:01

Comment: ARGON 100CC/MIN Pt PAN

Figure 37: TGA Curve for 400°C Distillation Residue D035-3

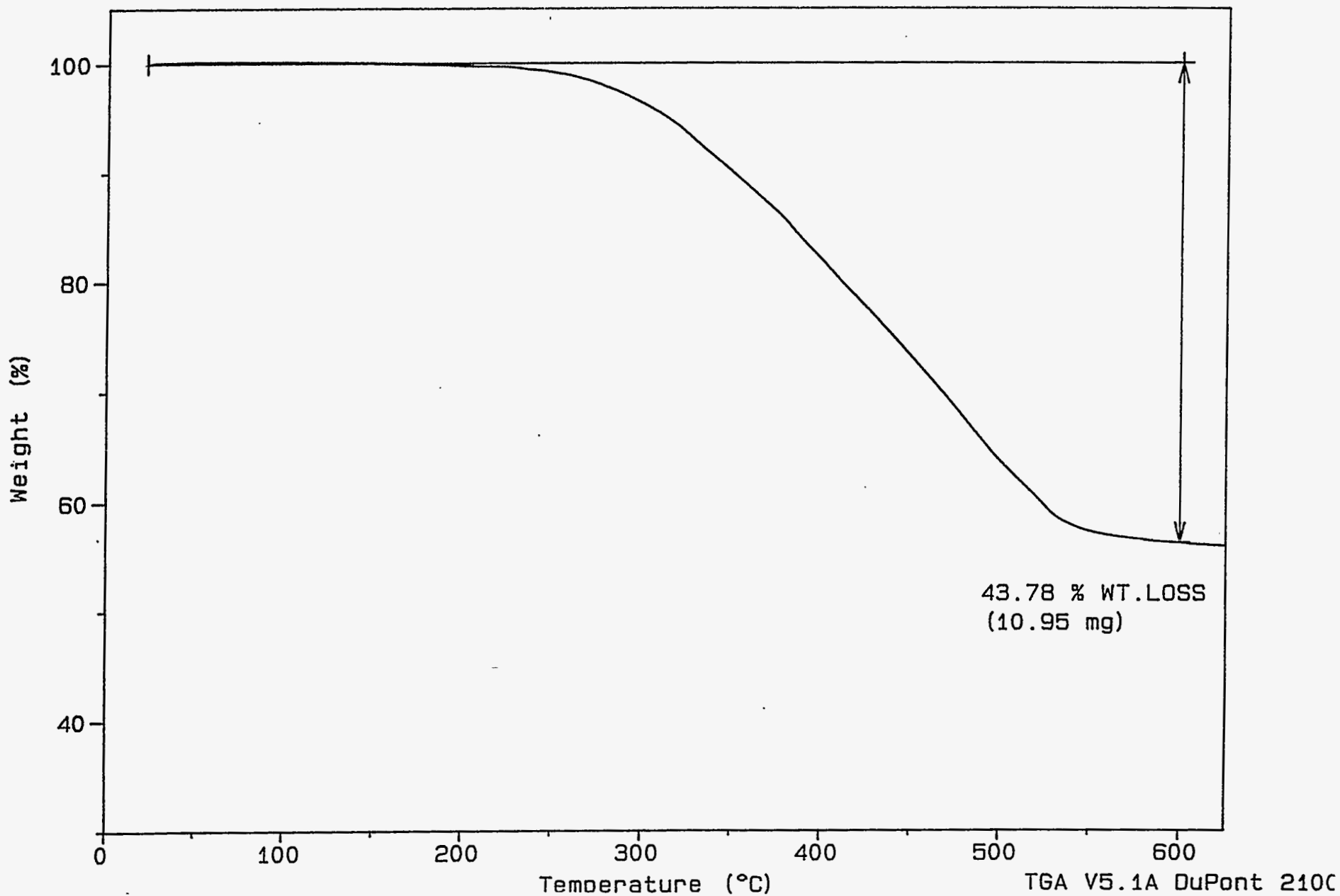


Figure 38

Annealed Mesophase Pitch from Sample A076

450°C - 2 Hr., Coal WVGS 13421

A076 450 - 2 HR (1342)



95091810-2-1 50X PL

500µm

50X, POLARIZED LIGHT

A076



95091810-2-2 250X PL

100µm

250X, POLARIZED LIGHT

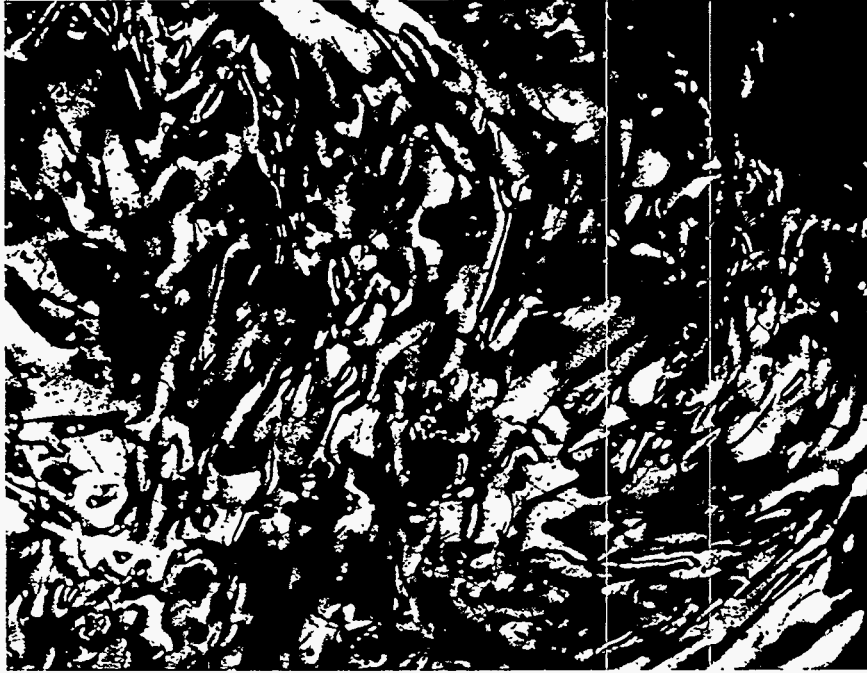
Figure 39

Annealed Mesophase Pitch from Sample A066

450°C - 1 Hr., Coal WVGS 13421

A066

450-1HR, 13421



95091810-1-1 50X PL

500μm

50X, POLARIZED LIGHT

A066



95091810-1-2 250X PL

100μm

250X, POLARIZED LIGHT

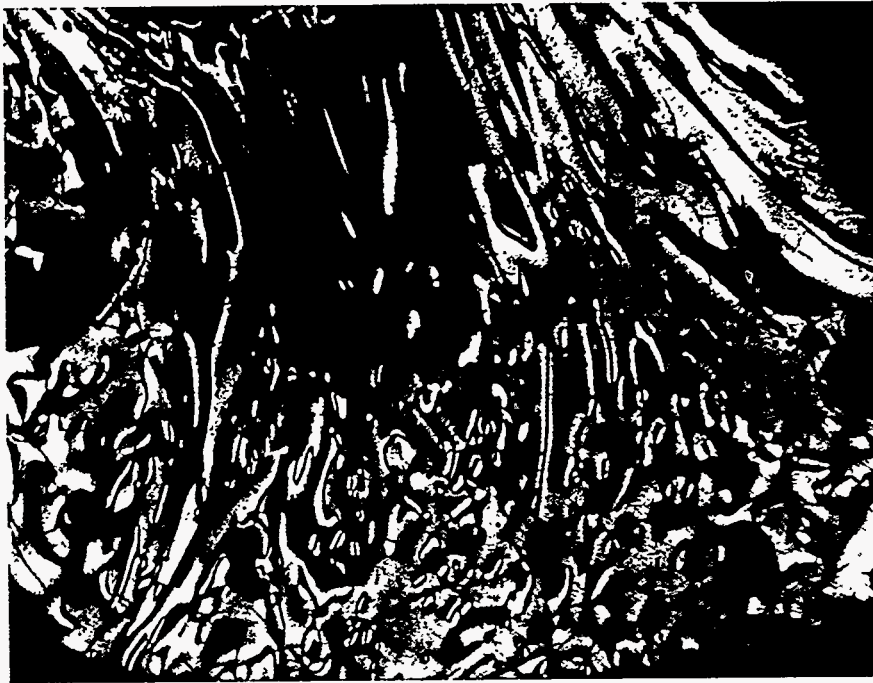


Figure 40

Annealed Mesophase Pitch from Sample A073

450°C - 2 Hr., Coal WVGS 13423

A073 450 - 2 Hr 13423



95091810-3-1 50X PL

500μm

50X, POLARIZED LIGHT

A073



95091810-3-2 250X PL

100μm

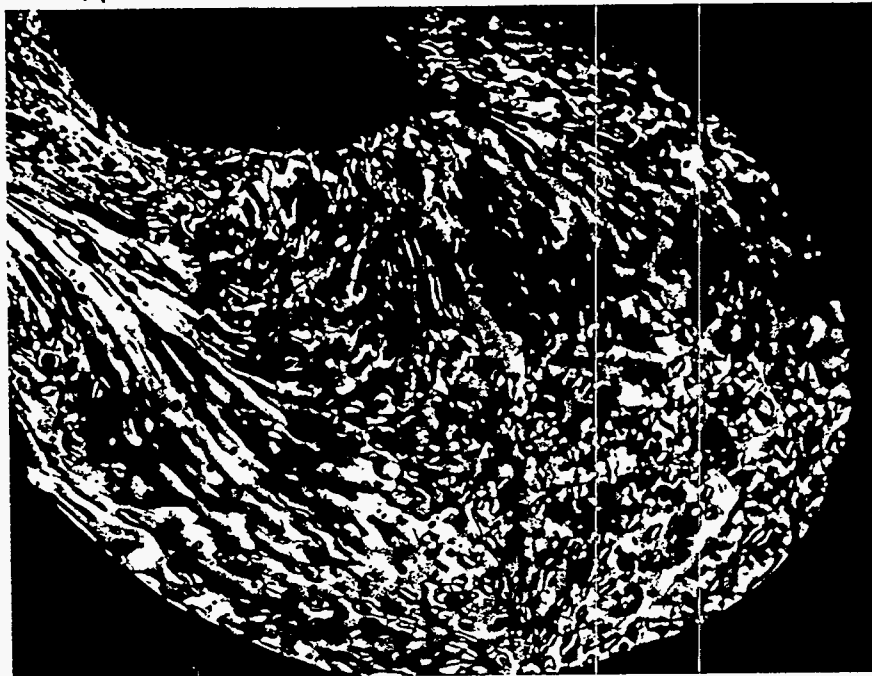
250X, POLARIZED LIGHT

Figure 41

Annealed Mesophase Pitch from Sample A075

450°C - 1 Hr., Coal WVGS 13423

A075 450 - 1 HR 13423

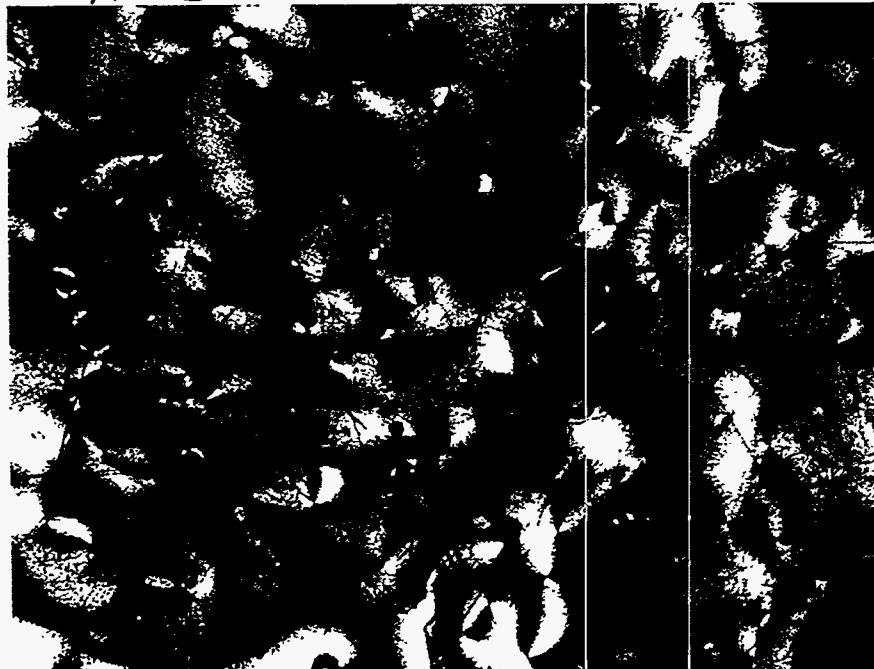


95091810-4-1 50X PL

500µm

50X, POLARIZED LIGHT

A075



95091810-4-2 250X PL

100µm

250X, POLARIZED LIGHT

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