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Modification of SHELL 405® Catalyst

Test Catalyst Preparation

AD 520319

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P.H. Williams

SHELL DEVELOPMENT COMPANY
A DIVISION OF SHELL OIL COMPANY
Emeryville, California

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April 1972

Final Report for Period February 1970 – December 1971
Contract F04611-70-C-0020

PREPARED FOR
Air Force Rocket Propulsion Laboratory
Director of Laboratories
Air Force Systems Command
United States Air Force
Edwards, California

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FOREWORD

This work was done at Shell Development Company, Emeryville, California for the Air Force Rocket Propulsion Laboratory under Contract F04611-70-C-0020, initiated 2 February 1970 and completed 31 January 1972. The Shell number for this report is S-14124.

The work under the contract was supervised by Dr. F. H. Williams. The experimental studies and catalyst preparations were carried out by Mr. Yosh Sakauye.

The work was conducted under the technical management of Capt. Douglas D. Huxtable and Lt. Ronald J. Meetin of the AFRPL/LKDP.

This report contains Classified information extracted from documents listed under References.

This technical report has been reviewed and is approved.

Ronald J. Meetin, 1st Lt. USAF
Project Engineer

ABSTRACT

This report describes a study carried out for the Air Force Rocket Propulsion Laboratory to develop a catalyst of the Shell 405 type with improved durability to repeated cold starts using monopropellant hydrazine. Catalysts that differed from standard Shell 405 Catalyst in respect to composition and properties of the support material and to the conditions of catalyst preparation, iridium content, and concentrations of certain normal trace impurities were prepared for testing at the AFRPL.

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MODIFICATION OF SHELL 405 CATALYST

(U) Introduction

The development of Shell 405 Catalyst made practical the use of hydrazine as a high energy monopropellant in thrust motors for space vehicles, and in gas generators for a variety of auxiliary and emergency functions. Over the years the inherent simplicity and other advantages of the hydrazine-catalyst system has led to growing use of Shell 405 Catalyst, notably in space applications. The present study was prompted in part by the possibilities of extending the uses of Shell 405 Catalyst and hydrazine into applications whose requirements would surpass the limits of reliable performance of the current catalyst, but that might be met by a longer-life or otherwise more durable modification.

The NASA-sponsored study1) which led to Shell 405 Catalyst was directed toward finding a catalytic agent which would initiate hydrazine decomposition, have short ignition delay, and multiple restart capability at low temperatures, and have good thermal and mechanical stability. Catalyst performance was measured by a liquid hydrazine activity test and short-duration test firings in a small reactor.

The present study, sponsored by the AFRPL, was directed toward improving the operating life of the catalyst, measured by its maximum repetitive cold start capability. The approach was primarily to test potentially more durable support materials and, simultaneously, to determine the effect on catalyst performance and life of variable support properties. The effects of varying process conditions, iridium metal content, and other factors were also examined. Catalyst samples designed to test the effects of these variations were prepared by Shell Development Company and were evaluated through engine and laboratory activity tests at AFRPL.

(U) Support Selection and Properties

The original study leading to Shell 405 Catalyst established through a comprehensive investigation of support materials that high-purity aluminas of high surface area afforded catalysts of the best all-around properties of activity, catalyst life, and physical stability. Harshaw AL-1404 T 1/8 alumina was the most satisfactory pelleted support, and Reynolds RA-1 activated alumina was the outstanding granular material for the preparation of a range of mesh sizes. The study also included an examination of a number of other refractory materials that might provide greater durability than the aluminas and indeed, a few appeared of promise. These were not proved out by firing tests, however.

In the present study, the substrate materials considered for evaluation were limited to alumina-based compositions because they showed promise for catalyst life improvement and, at the same time, afforded the best opportunity to examine the effect of varying support properties without major changes in composition. In any case, limitations of the study precluded a broad investigation of other types of support materials or the preparation of "tailored" materials in attempts to systematically and independently vary support properties. Test supports were therefore selected from the many commercial and developmental products that could be readily obtained.

Candidate supports were selected to provide finished catalysts with a range of surface area, pore volume, and pore size distribution as well as good retention of surface area at elevated temperatures. (Variations in bulk density and crush resistance were also of interest, but since bulk density is usually inversely dependent on porosity, and crush resistance is effected by porosity as well as particle shape and smoothness, selections were not made on the basis of these two properties.) Several alumina-silica supports were included because it has been shown that they retain high surface area at prolonged elevated temperatures.2,3) The base line support was the granular Reynolds RA-1 activated alumina4) used in standard Shell 405 Catalyst manufacture.

The following were selected for testing and comparison with the RA-1 as catalyst supports:

Esso Type Supports - granular aluminas containing approximately 6% SiO_2 and 5% of an alkaline earth oxide3)

Harshaw AL 1602 - 1/8" x 1/8" pellets of alumina containing 6% $SiO_2$5)

Reynolds "Strenghthened" RA-1 Alumina - an experimental granular alumina6)

Reynolds "Eta" Alumina - an experimental alumina, rich in Eta phase6)

Kaiser Chemical KA-201 - balls of activated alumina7)

Pechiney-Saint-Gobain Spheralite SAS 350 B - balls of alumina with 2-3% added $SiO_2$8)

These supports range in surface area from about 115 to 350 m^2/g and in pore volume by BET nitrogen adsorption from 0.22 to 0.45 ml/g. Bulk density ranges from 0.7 g/cc for materials of highest porosity to 1.05 g/cc for those of lowest porosity. Crush resistance is relatively high for all of the materials.

Physical properties and BET nitrogen adsorption (Adsorptomat) data for all test supports, including the RA-1 activated alumina, and the catalysts prepared with them are given in Table 1. Differential pore size distributions for all supports and catalysts are shown by the figures in Appendix III. These show the fraction of the specific pore volume per unit diameter plotted against pore diameter. The units of specific pore volume are microliters per gram and Angstroms for the diameter, hence the units microliter/g-angstrom for the ordinate of the plots.

The data and figures are of interest in that they show not only the wide range of surface area and pore volume of the supports (and catalysts), but, perhaps of more significance the pore size distribution. The uncalcined RA-1 activated alumina has the greater proportion of its micro pores in the diameter region below about 50 Angstroms, a proportion appreciably greater than for any of the other supports examined; the Pechiney SAS 350 B is the only other of the support approaching this distribution. In contrast, the pore distribution of the experimental Reynolds ETA alumina is preponderately well above 50 Angstroms and of much broader distribution. The data and figures also indicate the heat stability of the support by showing the effect of high temperature calcination on surface area and pore size distribution. The effect is notable with the RA-1 activated alumina which undergoes a large reduction in surface area on calcination at 900 C and a major shift of pore size to the region above 50 Angstroms diameter. Calcination of the RA-1 at lower temperatures has similar but lesser effects, as shown by other data in the table and by the figures. The corresponding changes on calcination of the silica-containing supports, (the Esso types and Harshaw AL 1602) were relatively small.

It was anticipated that the greater heat stability of the alumina-silica supports, as indicated by the nitrogen adsorption data, would be reflected in an increased stability and durability of the catalysts under test firing conditions.

From the data in the tables it will be noted that calcination had little or no effect on the pore volume of the calcined supports. This is interpreted as indicating that calcination causes small micro pores to coalesce into larger micro pores, which results in a reduction in pore surface area with little effect on total pore volume.

Macrostructures of the supports are indicated by micrographs of some of the catalysts prepared on them. Scanning electron micrographs, taken in the secondary electron mode at magnifications up to 30,000[9], display the micromorphology of external and fracture surfaces of some of the catalysts (Figures 1 and 2). These indicate that the RA-1 type alumina supports consist of conglomerates or clusters of pseudo-morphic crystal-like particles (reflecting their origin from the crystalline aluminum trihydrate Gibbsite), with openings like crevices or fissures throughout the structures. The micrograph at 30,000 times magnification seems to show fine pore openings on the surfaces. The Esso type and Harshaw supports appear to be amorphous-like materials, without the macro openings and passages of the RA-1 aluminas. Micrographs of the fired catalysts are very similar to those of the unused catalysts and show no changes in morphology. Catalysts prepared on the Kaiser and Pechiney supports were not examined by electron scanning.

The photographs in Figure 3 show the rounding and smoothing effect of the attrition treatment that is applied to standard RA-1 granular alumina before catalyst preparation. All of the test supports were similarly attrited before use.

(U) Process and Composition Variations

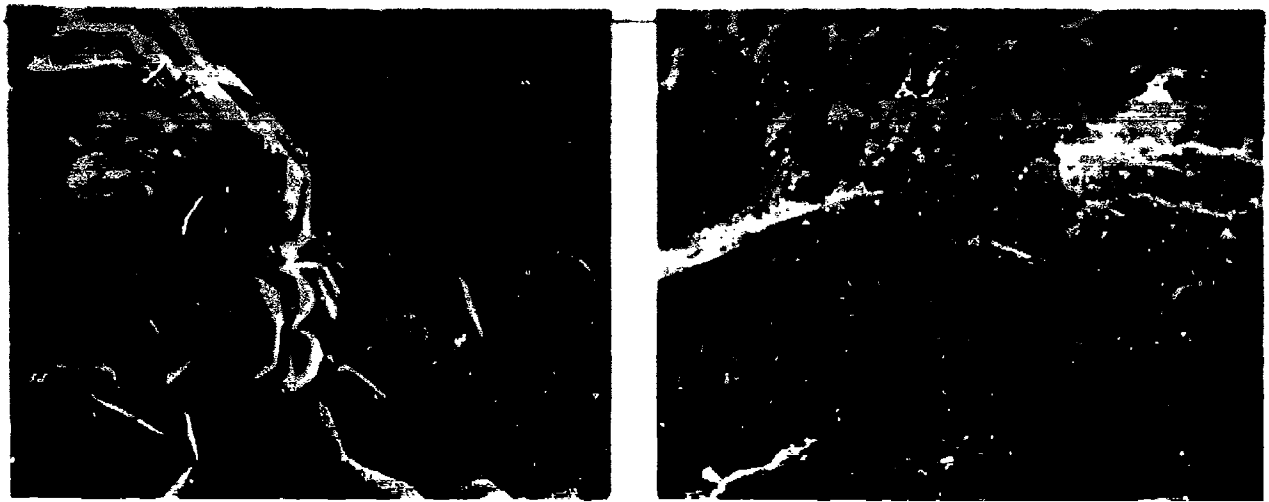
Processing studies were limited to variations of the standard catalyst manufacturing procedure, namely the preliminary support treatments of attrition and calcination, and the conditions of decomposition and reduction following impregnation of the support with the iridium salt. Composition studies, aside from the differences in support compositions already discussed, were limited to varying the iridium content and the concentrations of certain trace impurities in the finished catalyst. Except for the calcination treatments, the only support used was the standard RA-1 alumina.

These processing and composition modifications are discussed more specifically below under Catalyst Preparations.

(U) Catalyst Preparation

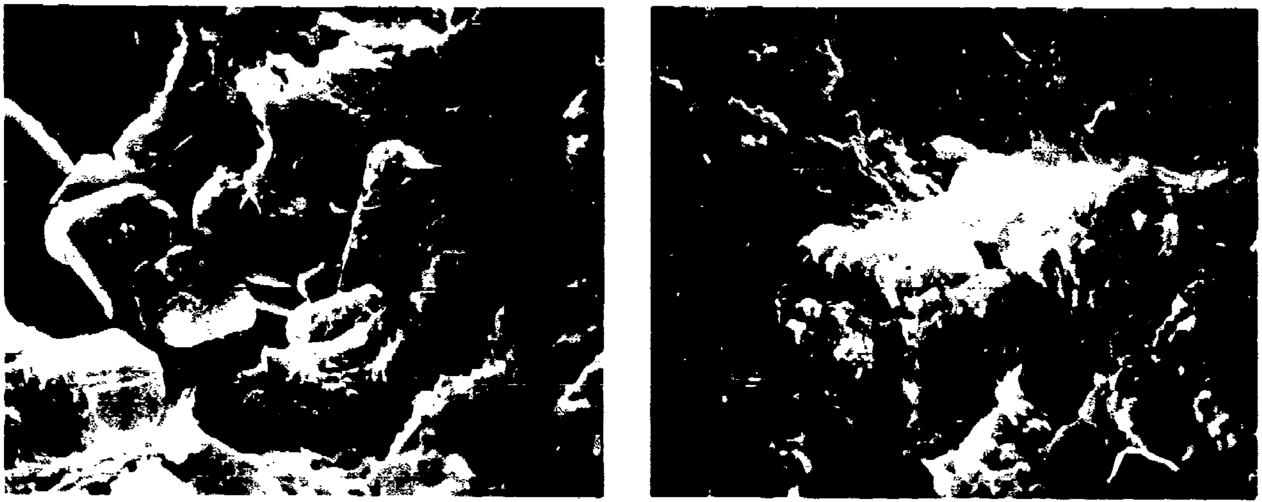
(U) All of the test catalysts (except the plant standard product) were prepared in the laboratory and in accordance with established Shell 405 Catalyst manufacturing procedures and quality control, except for modifications in apparatus and conditions that were necessary for small scale preparations, and processing and raw material changes that were, in essence, specific objectives of the test program. These modifications are described in Appendix I - Experimental.

Figures 1 through 3 follow



Reynolds RA-1 Alumina (1)
(Standard Shell 405 Catalyst)

Esso 504 Type (8)



Reynolds RA-1 Alumina (3)
(Calcined at 900°C)

Harshaw Al-1602 (10)

Reynolds RA-1 Strengthened Alumina (13)

Reynolds Eta Alumina (14)

Figure 1. FRACTURE SURFACES OF CATALYSTS ON TEST SUPPORTS
Scanning Electron Micrographs (3000X) - Numbers Reference Table 1

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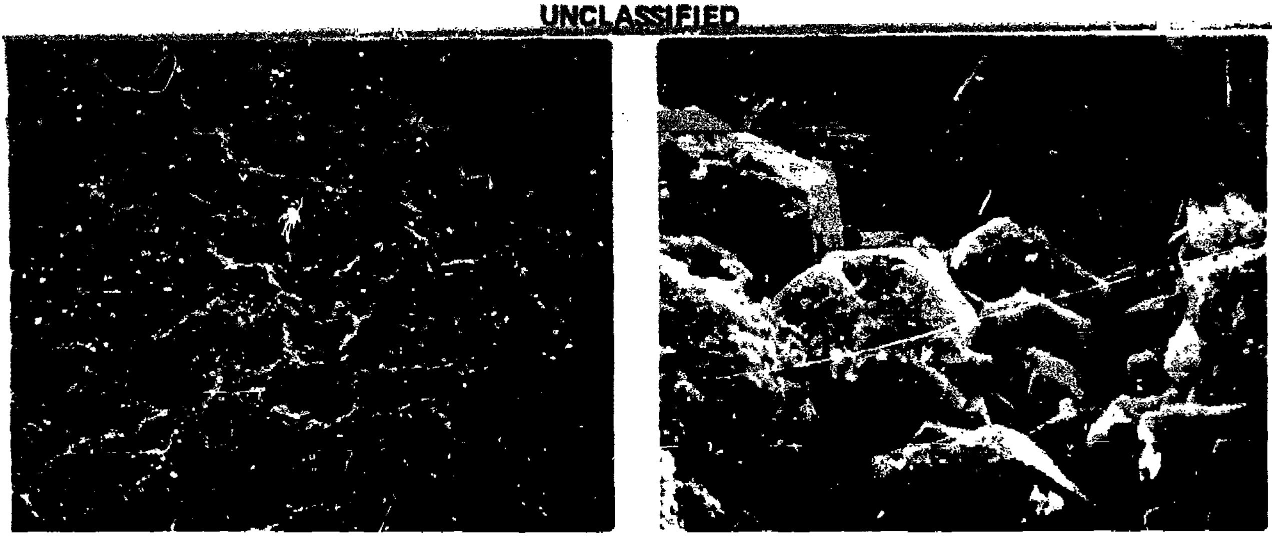
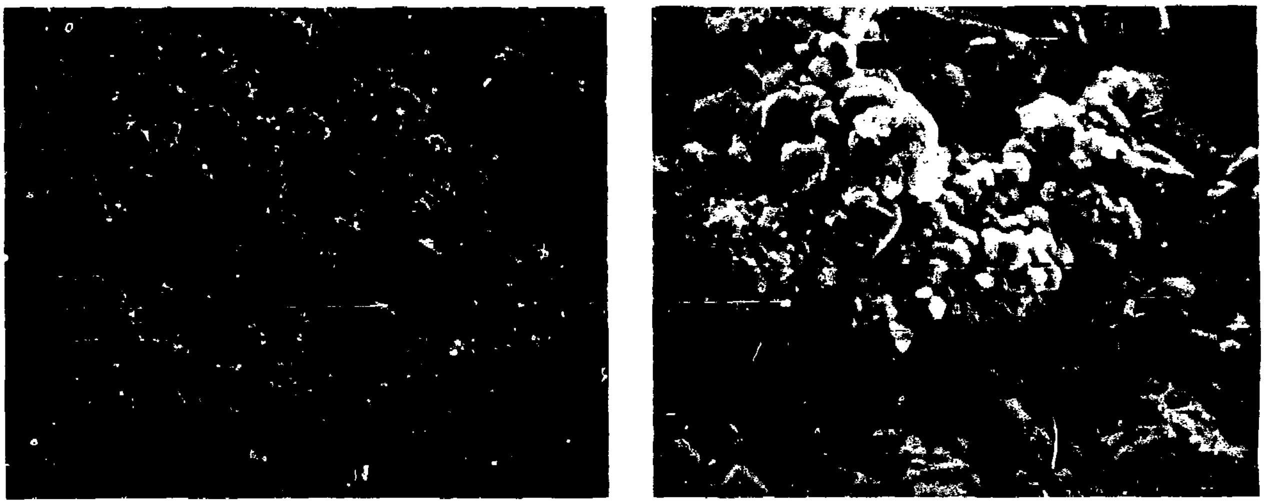


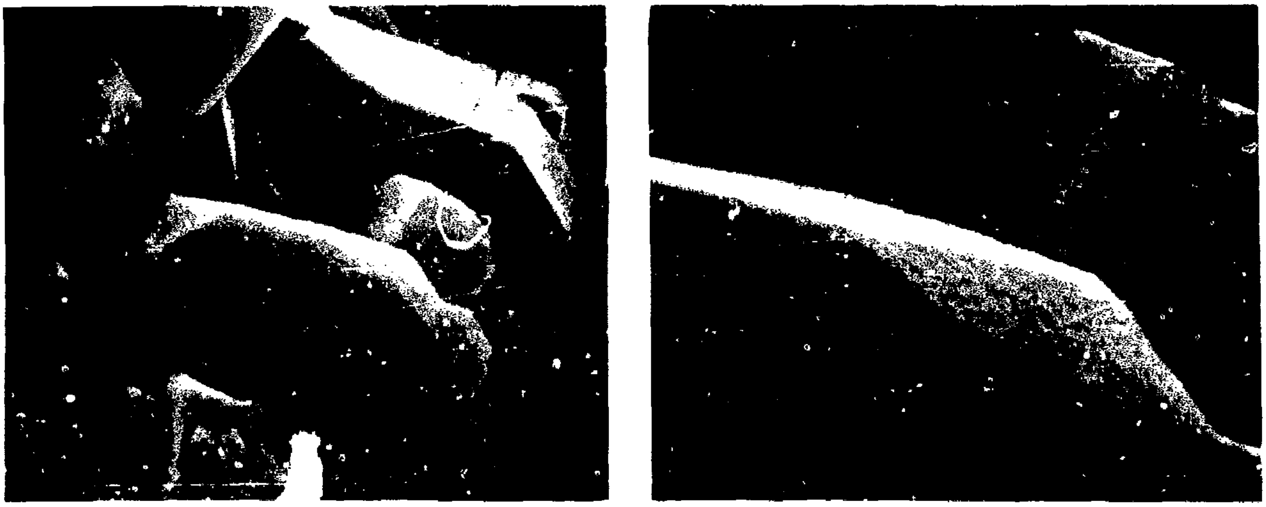
Exterior Surface *(300X)*

Exterior Surface *(3000X)*



Fracture Surface *(300X)*

Fracture Surface *(1,000X)*



Fracture S[illegible]ce *(10,000X)*

Fracture Surface *(30,000X)*

Figure 2. STANDARD SHELL 405 CATALYST (ON REYNOLDS RA-1 ALUMINA)
Scanning Electron Micrographs

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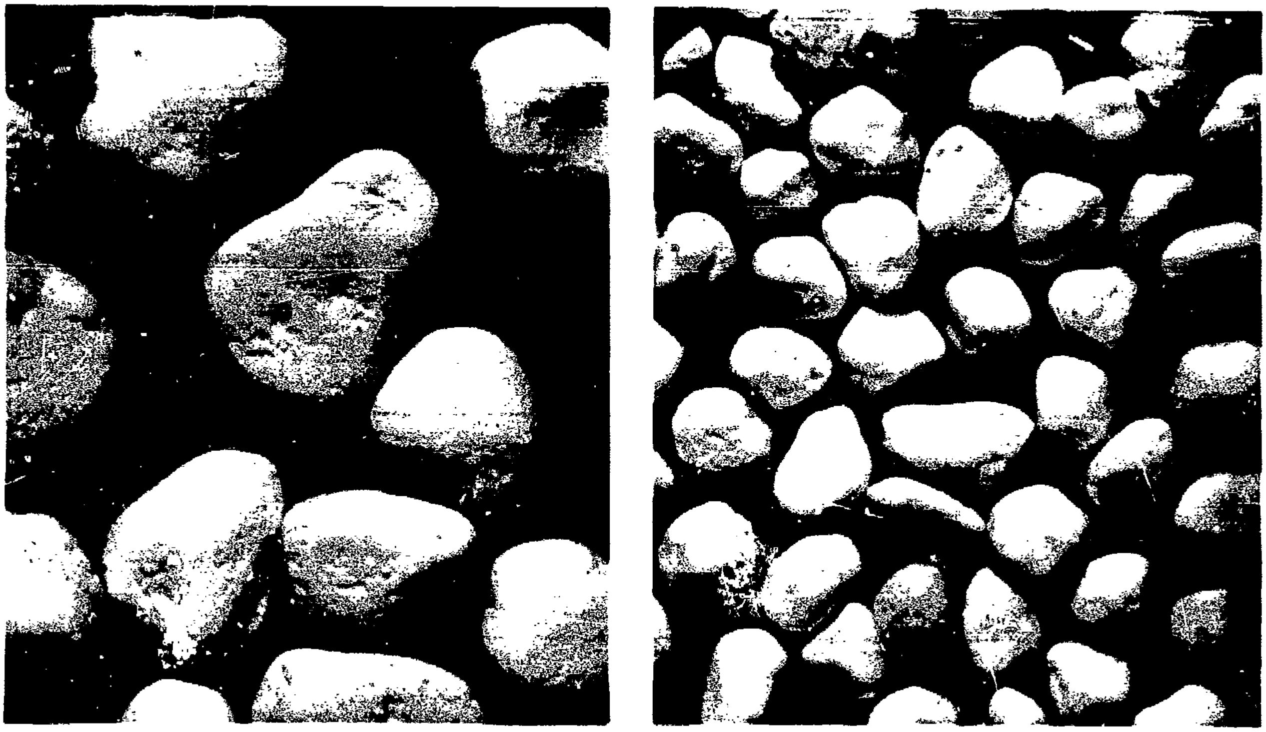
14-18 Mesh – As Ground

14-18 Mesh – 50% Attrited



14-18 Mesh – 90% Attrited

25-30 Mesh – 90% Attrited

Figure 3. EFFECT OF ATTRITION OF REYNOLDS RA-1 ALUMINA GRANULES
Magnification: ca. 18X

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(U) For Support Evaluation

All of the supports were attrited by the conventional water-attrition process prior to catalyst preparation. This treatment eliminates soft and easily fractured granules and removes sharp, rough edges. This gives smoother, partially rounded, and more consistent strength granules which results in catalysts that pack more readily and densely and have less tendency to abraid and fragment in use. The two spherical supports were attrited lightly to reduce their size. Several supports were also given calcination treatments to preshrink and otherwise stabilize them. Properties of the catalysts prepared are summarized in Table 1.

The Reynolds RA-1 Alumina was used in four laboratory preparations: a conventional preparation to compare laboratory- to plant-prepared catalyst and to catalysts prepared on other supports; a second catalyst on calcined support to determine the effect of heat-stabilization; a third catalyst on a support that had been severely attrited (to 90% weight loss) to give a rounder and harder substrate; and a fourth preparation to determine the effect of both severe attrition and finer mesh size on catalyst performance.

The Esso-type supports, containing silica and an alkaline earth oxide, were previously shown to have excellent surface area retention at high temperatures. Four test catalysts were prepared, in two mesh sizes, on calcined (900 C) and uncalcined Esso supports.

Harshaw AL 1602 is a commercial alumina-silica, calcined at 600-700 C. Catalysts prepared on this support were previously shown to have excellent surface area retention under high temperature use.2) For this program, catalyst was prepared on the attrited support, on support that was calcined at 900 C, and on support that had been treated with aqueous sucrose and calcined at 1100 C in an attempt to make a stable (high alpha-phase) alumina with moderately high surface area.

The Reynolds "strenghthened" RA-1 Alumina was an activated alumina especially treated by Reynolds to strengthen or harden it. This support was calcined at 900 C before use in catalyst preparation.

Reynolds "Eta" alumina was reported by Reynolds to have exceptional surface area stability over prolonged periods at 700 C. The support sample, as received, had been calcined at 700 C for 48 hours. One test catalyst was prepared.

The Kaiser KA-201 active alumina was used as support to test the effect of spherical shape on catalyst durability. The Pechiney Spheralite SAS 350 B containing 2-3% silica, was claimed to have good surface stability at temperatures as high as 870 C and, also, was another example of a ball-shaped support. A catalyst was prepared with each material.

(C) (U) For Effect of Process and Composition Variations

A second series of catalyst was prepared to test the effect on catalyst performance of changes in iridium content, process conditions, and the presence of certain impurities. The standard RA-1 alumina, granular support used for all preparations, was attrited to fifty percent weight loss. Specific variations in catalyst preparation are described below, and properties of the resultant catalysts are given in Table 2.

(U) Iridium Content. The standard catalyst contains from 31 to 33 weight percent iridium. The early catalyst studies had indicated that this was the approximate minimum amount of iridium on alumina to give catalysts that, in a laboratory test engine with neat hydrazine, would give at least ten cold (2-4 C) starts, each within an arbitrary 100 millisecond response time. For the present study, a series of catalyst containing from about ten to fifty percent iridium was prepared to determine the optimum metal content for satisfactory catalyst activity and maximum cold start durability.

(C) Hydrogen Reduction Temperature. The temperature of hydrogen reduction of the iridium salt-impregnated support affects surface area and active metal area of the finished catalyst and thus its initial activity: The higher the temperature, the lower the surface and active metal areas. It was expected that reduction could be carried out at a temperature which, while appreciably diminishing initial activity, would also result in a "preshrinking" and "stabilization" of the catalyst. This, paralleling the intent in post-shrinking the catalyst support by calcination, might result in a catalyst with longer active life under repetitive cold start operating conditions and minimum loss through abrasion and fracturing.

(C) Catalysts were accordingly prepared, to determine temperature effects, at reduction temperatures bracketing the standard temperature condition. The higher temperature chosen (800 C) and treatment duration approached those sometimes used in preparing pre-calcined support for otherwise standard catalyst. As shown by the data in Table 2, the higher temperature reduction resulted in a catalyst of markedly lower surface area and hydrogen chemisorption values relative to catalyst prepared under standard conditions. The values of these properties were roughly the same as for the test sample prepared on pre-calcined support (Catalyst 11724-64, Table 1).

(C) Salt Decomposition Conditions. In the preparation of standard catalyst, the impregnated iridium salt is partially decomposed by passing through the support a stream of heated dry air. Two catalysts were prepared to determine the effect of substituting either nitrogen or moist air for dry air in this process step; a third catalyst was prepared under standard conditions with dry air for comparison. Examination of the data in Table 2 shows that these processing variations resulted in catalysts that did not differ significantly in the measured properties.

(U) Effect of Added Impurities. Sulfate ion, and sodium, iron, and other metal impurities are normally found in standard catalysts in amounts of hundredths percent or less. Effects have not been observed on catalyst performance of impurities at these levels; however, for quality control, very low maximum levels have been set and maintained by analyses of raw materials and finished catalysts. The contents of sulfate, sodium, and iron are of special interest because these impurities have shown an appreciable variability in the raw materials used. Lead has been an insignificantly low impurity in the iridium salt used, but this element may be present in significant amounts in some grades of iridium metal, and its effect on catalyst activity and life was therefore of interest.

The impurities were added as suitably soluble salts to the supports in the course of catalyst preparation at concentration levels appreciably above the amounts normally found or, by specification, permitted in the raw materials or finished catalysts. The impurity contents and other properties of the test catalysts prepared are given in Table 2. The catalyst containing lead had a lower than normal surface area, and the sample impregnated with sodium and sulfate ions had an unusually low active metal area by hydrogen chemisorption measurement.

(U) Catalyst Analyses and Properties

Analytical and other data for the test catalysts are included in Tables 1 and 2, and plots of Adsorptomat data appear in the Appendix. A summary of test methods is also in the appendix.

BET nitrogen adsorption data show, as expected, that the catalysts have lower pore volumes and surface areas than their support materials. The pore volumes of catalysts and supports have an approximately inverse relationship to their densities; the surface areas of the catalysts are not only lower than the supports because of greater densities, but also in most cases because of effects of process treatments. The catalyst prepared at high hydrogen reduction temperature has a markedly lower surface area than catalyst prepared at standard reduction temperature. (The similar effect of high temperature calcination on support surface area has already been noted.)

Processing conditions and composition changes effected the active metal area, as determined by hydrogen chemisorption measurements. Of particular interest are the increasing chemisorption values with increasing iridium content (and catalyst densities) up to about 30 to 40 percent iridium, and the sharply lower value for the catalyst containing 50 percent iridium. The chemisorption value was also below standard for the catalyst reduced at high temperature and the catalyst containing added sulfate (and sodium) impurity. The variations in impregnated support decomposition techniques did not have a notable effect on chemisorption.

As previously discussed, electron scan micrographs of catalysts on several of the test supports are shown in Figures 1 and 2.

(U) Conclusions and Recommendations

A. Conclusions

1. The physical data in Tables 1 and 2 demonstrate the feasibility of empirically varying catalyst properties by appropriate selection of support materials, and by modifying conditions of catalyst preparation and active metal content. Catalysts ranging considerably in surface area, pore volume and pore volume distribution, and hydrogen chemisorptivity were obtained with use only of alumina and alumina-silica supports, and without major change in the standard catalyst preparation procedure. Similar or even greater variations in physical properties might be realized with supports of other elemental compositions.

2. Conclusions as to the relationship of these physical properties to catalyst activity and life depend on the results of the activity and firing tests that are being carried out at the AFRPL.

B. Recommendations

1. The physical properties of the supports and catalysts may not be the only, or even the most significant, characteristics which determine catalyst

life. In reference particularly to alumina support materials, other factors such as the following may effect the durability of the catalyst under firing conditions:

a. Support origin (there are several natural and synthetic structural varieties of aluminas).

b. Support microstructure (amorphous; crystalline; pseudomorphs of crystal forms).

c. Support manufacturing steps (i.e., crystallization; gel dehydration; precipitation; extrusion; pelletization).

d. Trace impurities in the support (e.g., calcium, silica, sodium).

e. Support pretreatments (e.g., with a silicate to increase surface area retention).

f. Catalyst formulation method (impregnation of support or alternative techniques of combining support and active ingredients).

g. Other catalyst preparation conditions (e.g., pH and concentration of salt impregnant, use of non-aqueous solvents).

Alumina-based catalysts should be prepared and test fired to determine the effect and importance of these factors on catalyst life.

2. Selected materials of other elemental compositions should be evaluated as catalyst supports.

Table 1. COMPOSITION AND PHYSICAL DATA - EVALUATION OF SUPPORTS

Support/Catalyst			Adsorptomat Data						Hydrogen Chemi-sorption µ Moles/g	Bulk Density g/cc	Crush Strength % Survival
Description	Treatment (a)	Iridium %w	Surface Area m²/g	Pore Vol. cc/g	Vol. Average Diam., Angs b)	Area Average Diam., Angs	Vol. Median Diam., Angs	Area Median Diam., Angs			
Reynolds RA-1, 14-18 1. Catalyst 12-MEM-403	- (Plant standard)	- 31.7	253 108	0.236 0.12	103.8 102.1	37.3 44.7	31.6 43.1	26.9 31.7	- 403	1.01 1.58	97.7 -
2. Catalyst 11724-51	(Laboratory standard)	31.2	133	0.13	107.4	40.1	36.1	28.7	470	1.52	-
Reynolds RA-1, 14-18 3. Catalyst 11724-64	Calc 900°C -	- 31.7	96 62	0.235 0.13	162.1 159.7	94.3 87.1	101.8 94.6	71.2 64.1	- 343	1.01 1.55	94.2 -
Reynolds RA-1, 14-18 4. Catalyst 11724-60	90% attrited -	- 31.6	263 122	0.23 0.13	91.4 106.4	36.2 44.0	31.0 39.4	26.5 31.2	- 454	1.02 1.55	98.8 -
Reynolds RA-1, 25-30 5. Catalyst 11724-28	90% attrited -	- 31.4	258 133	0.22 0.125	84.4 90.4	34.6 38.2	30.4 34.9	25.8 28.0	- 382	1.04 1.61	99.9 -
Esso 500, 16-18 6. Catalyst 11724-56	- -	- 32.2	193 143	0.36 0.23	105.0 106.1	73.6 65.1	79.2 73.9	61.2 50.3	- 485	0.90 1.40	92.7 -
Esso 500, 14-18 7. Catalyst 11724-57	Calc 900°C -	- 32.0	130 123	0.33 0.23	116.8 116.7	85.5 75.5	86.4 84.6	71.7 60.0	- 409	0.90 1.40	98.2 -
Esso 504, 20-30 8. Catalyst 11724-12	- -	- 31.9	223 147	0.39 0.23	101.7 102.9	72.2 66.0	75.9 74.2	58.5 50.9	- 450	0.81 1.27	91.6 -
Esso 504, 20-30 9. Catalyst 11724-34	Calc 900°C -	- 31.0	190 134	0.36 0.21	105.9 104.1	79.0 69.5	86.5 83.7	67.1 51.5	- 424	0.82 1.25	92.8 -
Harshaw Al-1602, 14-18 10. Catalyst 11724-7	- -	- 31.7	240 163	0.44 0.27	114.5 125.0	73.9 68.1	81.4 82.4	56.1 46.7	- 504	0.76 1.20	90.6 -
Harshaw Al-1602, 14-18 11. Catalyst 11724-13	Calc 900°C -	- 31.6	194 137	0.43 0.25	128.3 124.1	85.9 73.7	96.6 90.1	67.5 51.8	- 400	0.79 1.32	95.1 -
Harshaw Al-1602, 14-18 12. Catalyst 11724-48	Calc 1100°C -	- 31.9	161 105	0.36 0.23	138.4 138.9	91.0 87.4	104.1 105.7	71.2 65.7	- 284	0.94 1.55	91.4 -
Reynolds RA-1 Strengthened, 14-18 13. Catalyst 11724-47	Calc 900°C -	- 3[illegible].4	78 48	0.24 0.16	229.7	158.7	172.4	102.1	243	1.[illegible]0 1.19	97.6 -
Reynolds "Eta" Alumina, 14-18 14. Catalyst 11724-40	- -	- 31.[illegible]	117 90	0.43 0.25	238.0 221.4	157.1 116.7	186.3 165.7	123.0 72.2	- 307	0.75 1.19	94.1 -
Kaiser KA-201, 14-18 15. Catalyst 11724-106	- -	31.1	349 163	0.455 0.27	116.1 135.5	54.1 68.8	57.0 76.6	34.7 45.9	- 503	0.75 1.18	98.1 -
Pechiney SAS 350 B, 14-18 16. Catalyst 11724-107	- -	32.5	273 158	0.43 0.26	96.0 103.1	61.8 65.8	65.4 73.0	48.0 51.8	- 497	0.90 1.38	99.5 -
Reynolds RA-1, Strengthened	(as rec'd)	-	150	0.29	125.0	74.3	73.7	59.6	-	1.00	97.3
Reynolds RA-1, 14-18	Calc 500°C	-	209	0.262	115.9	48.4	42.2	33.3	-	-	-
Reynolds RA-1, 14-18	Calc 600°C	-	167	0.259	123.8	59.4	53.8	42.3	-	-	-
Reynolds RA-1, 14-18	Calc 700°C	-	143	0.246	129.9	66.9	61.9	49.7	-	-	-
Reynolds RA-1, 14-18	Calc 800°C	-	108	0.240	148.8	81.7	75.9	61.5	-	-	-
Reynolds RA-1, 14-18	Calc 900°C	-	86	0.245	184.3	103.9	109.4	75.8	-	-	-

a) Non-standard treatments applied to supports.

Table 2. COMPOSITION AND PHYSICAL DATA - EVALUATION OF PROCESS AND COMPOSITION VARIABLES

Catalyst[a]		Process[b] Variation	Iridium %	Adsorptomat Data						Hydrogen Chemi-sorption μ Moles/g	Bulk Density g/cc
Item No.	Reference			Surface Area m^2/g	Pore Vol. cc/g	Vol. Average Diam., Angs	Area Average Diam., Angs	Vol. Median Diam., Angs	Area Median Diam., Angs		
17.	11724-68	(Standard lab prep.)	31.6	126	0.135	112.7	42.7	37.9	30.3	468	1.54
18.	11724-78	Impregnation	10.2	183	0.201	95.2	42.8	38.8	30.8	211	1.18
19.	11724-79	Impregnation	19.6	148	0.168	107.1	44.3	39.9	30.5	360	1.34
20.	11724-80	Impregnation	39.1	105	0.121	111.2	45.3	41.0	30.3	445	1.74
21.	11724-108	Impregnation	49.5	77	0.093	122.7	49.8	52.0	31.7	305	2.11
22.	11724-94A	Red'n to 370°C	30.2	131	0.121	92.2	39.3	34.0	28.7	508	1.63
23.	11724-94B	Red'n to 800°C	31.9	77	0.126	124.2	64.4	65.5	46.8	300	1.55
24.	11724-95	Dec. in N_2	31.4	116	0.125	109.4	44.0	37.2	31.1	488	1.56
25.	11724-97	Dec. in dry air	31.3	114	0.124	106.9	44.1	37.9	31.5	425	1.58
26.	11724-98	Dec. in air/ H_2O	31.0	110	0.123	104.6	44.6	39.3	31.5	408	1.60
27.	11724-88	1.0% SO_4 0.16% Na	31.0	119	0.125	137.9	42.2	37.4	28.1	242	1.58
28.	11724-89	0.4% Fe	31.0	125	0.135	129.2	43.1	39.6	29.5	432	1.50
29.	11724-90	0.15% Pb	31.1	98	0.132	178.2	54.4	46.6	32.0	498	1.57

a) All catalyst on standard Reynolds RA-1 alumina, 14-18 mesh, 50% attrited, (see Table 1 for support data).
b) Non-standard process variation.

APPENDIX I

Experimental

(U) Raw Materials

Physical properties of the test support materials are included in Table 1. The standard support, Reynolds RA-1, alumina, is a high purity alumina analyzing in percent as follows: SiO_2, 0.06; CaO, < 0.01; Na_2O, 0.5; TiO_2, 0.005; Fe_2O_3, 0.03; SO_4, 0.03; L.O.I., 6.24; Al_2O_3, 93.1 (by diff.). Sodium content after water-washing: 0.06%.

The chloriridic acid (iridium tetrachloride), supplied by Matthey Bishop Inc., was high purity material containing: < 0.2% SO_4; 0.04% Na; 0.02% Fe; and 0.003% Pb.

(C) (U) Support Treatments

(U) Attrition - All supports were attrited by standard methods. The new supports were attrited approximately to the roundness and smoothness of the standard "50% attrited" RA-1 alumina. Samples of RA-1 alumina were also attrited to 90% sieved weight loss.

(U) The ball-shape Kaiser KA-201 and Pechiney SAS 350 B were attrited in water to reduce them to 14-18 mesh. KA-201, sieved to 12-18 mesh range and attrited for 24 hours, gave 78% recovery as 14-18 mesh material. SAS 350 B, sieved to 12-18 mesh range, gave 82% in the 14-18 mesh range after 24 hours of attrition. Both supports retained their spherical shape during attrition.

(C) Calcinations - These were carried out by heating the attrited supports in an open porcelain dish in a muffle furnace. The supports were held for one hour at the temperatures given in Table 1.

(C) A sample of Harshaw AL 1602 was soaked in 50% aqueous sucrose at 80 C, heated in a pyrex tube at 500°C then in a Vycor tube at 1100 ± 20°C, with nitrogen gas flowing through the bed in each case. The support was then calcined at 700 ± 20 C in a porcelain dish in the muffle furnace.

(U) Properties of the attrited and calcined supports are given in Table 1.

(C) Catalyst Preparation on Test Supports

Catalyst preparation consists essentially of three successive steps:

1. The support is impregnated with aqueous iridium salt and then heated to decompose the salt into a water-insoluble form. This step is repeated many times and until the desired amount of iridium has been adsorbed.

2. The impregnated support is treated with gaseous hydrogen to convert the iridium to a metallic or catalytic state.

3. The material is exposed to oxygen under controlled conditions to produce a catalyst which is stable to air under ambient conditions.

The standard laboratory preparative procedure is the same as the plant procedure except for the following differences:

a. The salt decomposition step is carried out by drying the impregnated support for ten minutes with a heated air gun, followed by heating 100 g quantities or less in an open six inch diameter petri dish to about 380 C over a 45 minute period on a hot plate. (In the plant, the support is contained in a vertically mounted funnel-like vessel, open at top and bottom, through which is passed heated air.)

b. In the reduction step, in which the material is contained in a vertical pyrex tube (approximately 1" I.D. x 12" length) heated in an electrical furnace, hydrogen flow is started at a heated bed temperature of 150 C, and the reduction reaction progresses at 450-500 C through the tube within about 15 minutes; hydrogen flow is continued an additional 15 minutes while maintaining the bed temperature at about 500 C and for a further period while the catalyst bed is allowed to cool. (Plant preparations, using a larger tube and larger catalyst quantities, require about an hour for reduction, and hydrogen flow is continued for additional two hours at 500 C.)

A total of sixteen catalyst samples of 50 to 85 g were prepared on the seven support materials. The preparative method, after support pre-treatments, was identical for all samples except that the number of impregnation cycles required to give 31-33% iridium content varied from 12 to 19 in correspondence with increasing pore volume of the supports. The finished catalysts were sieved to mesh range and analyzed. Analytical data are given in Table 1.

(C) (U) Other Catalyst Preparations

(U) All other catalysts were prepared with attrited RA-1 alumina with the variations in process conditions described below. The properties of the test catalysts are given in Table 2.

(C) Iridium Metal Content - Catalysts of approximately 10, 20, 39 and 50 percent iridium metal content were prepared on water-attrited RA-1 alumina by the standard procedure, except that 7, 11, 26 and 38 impregnation/decomposition cycles were required in order to obtain the respective metal contents. (Standard catalyst of 31-33% iridium requires 18 to 20 cycles.)

(C) Hydrogen Reduction Conditions - In preparation of Catalyst 11724-94A, reduction was initiated at about 75 C, but proceeded exothermically at about 370 C and was complete within 15 minutes. For Catalyst 11724-94B, reduction was initiated at 150 C and maintained at about 450 C for fifteen minutes; treatment with hydrogen was continued for one hour, maintaining a catalyst bed temperature of about 800 C by external heating.

(C) Salt Decomposition Conditions - In preparation of Catalyst 11724-95, the iridium salt on RA-1 support was decomposed after each cycle by passing heated nitrogen through the bed of material contained in a pyrex tube; in preparation of Catalyst 11724-98, the heated gas consisted of moist air prepared by bubbling air through water at ambient temperature. (The laboratory apparatus was modified for these treatments by addition of the pyrex tube in place of a petri dish. Since the modification itself might affect catalyst performance and properties, a laboratory standard catalyst, 11724-97, was prepared in the modified apparatus, using dry air as the decomposing gas for comparison with the previously-prepared laboratory standard.)

(U) Added Impurities - Three catalysts were prepared with sodium and sulfate ions, iron and lead as added impurities at levels above those normally encountered in the standard catalyst.

(C) Catalyst 11724-88 - This catalyst was prepared in the conventional manner through all of the impregnation-decomposition cycles. On completion of the final cycle, 34.1 g of unreduced catalyst was soaked for 15 minutes in 32.0 ml of 1.0 M aqueous sodium sulfate. It was then dried under house vacuum for 15 minutes (removing 17 g of solution) and reduced and oxidized in the usual fashion. Analysis of the catalyst by x-ray diffraction showed 1.0% sulfate content; sodium by photometric analysis was 0.16%.

(U) Catalyst 11724-89 - To the iridium salt impregnation solution was added 8.0 cc of 1.0 M aqueous ferric chloride. The catalyst was prepared in the usual manner. Analysis of the finished catalyst by x-ray fluorescence showed 0.4% Fe.

(U) Catalyst 11724-90 - To the iridium salt impregnation solution was added 1.0 ml of 1.0 M aqueous $Pb(NO_3)_2$. The catalyst was prepared in the usual manner. Analysis showed that it contained 0.15% lead.

APPENDIX II

Test Methods

(U) Pore Volume Distribution

Pore volume distributions and surface areas are calculated from nitrogen adsorption isotherms measured with the aid of volumetric gas and sorption apparatus, the Aminco "Adsorptomat".10) This instrument, which is based on the design of Ballou and Doolen11) automatically measures the amount of nitrogen adsorbed by a weighed sample at -196 C as a function of relative pressure over the range 0.01 to 0.995. The isotherm data are reduced by the method of Barrett, Joyner and Halenda12) as modified by the Emig and Hoffman13) to obtain a cumulative pore volume distribution over the range of 16 to 850 angstroms. Volumes of pores between various size boundaries are calculated and plotted as functions of the average size. Several mathematical properties are computed for the distributions of both the volumes and areas of pores with respect to size. These include the weighted average, where the weighting factor is the volume or area of pores having diameters in a certain narrow range. The median value is the diameter exceeded by that for pores containing half the volume or area of the total sample. The computations and plot are made with the aid of a computer. Many assumptions are involved in this and other methods for the determination of pore volume distribution in this pore size range and no general statement can be made about precision and accuracy.

(U) Surface Area

Surface areas are determined from the amounts of nitrogen adsorbed (using the Aminco Adsorptomat) by the sample at -196 C at several relative pressures in the range 0.05 to 0.3. The least squares estimates of the parameters of the Brunnauer-Emmet-Teller14) equation which represents these data are found numerically and used to calculate the surface area of the sample. Precision of the surface area values is ± 2 percent.

(U) Hydrogen Chemisorption

The amount of hydrogen chemisorbed by a weighed sample is measured by the dynamic method of Nelson and Eggertsen.15) The apparatus used consists of a sample cell, thermal conductivity bridge, recorder and associated equipment as needed to pass gas over the sample under various conditions. The sample is reduced in hydrogen at 500 C and then cooled to 0°C in an argon stream containing 1 percent hydrogen while the composition of the exit gasis recorded. Hydrogen is taken out of the gas stream by chemisorption in this step. After the chemisorption is complete, as shown by the return of the gas composition to its original level, the sample is heated to 500 C to release the chemisorbed hydrogen. The amount evolved is measured by referring the area under the peak in the composition-time curve to a calibration curve prepared from areas obtained by the addition of known amounts of hydrogen to the gas stream. The precision is ± 5 percent. The accuracy has not been established.

(U) Crush Strength

A ten gram-quantity of support or catalyst is pouredinto a heavy-walled stainless steel cylinder of internal dimensions 0.875-inches diameter and 1.5-inches depth. A stainless steel plunger 3 inches in length and machined to smooth-sliding diameter is inserted into the cylinder and the assembly is placed in a hand-operated hydraulic press. A force of 375 pounds (625 psi) is applied, held for 10 seconds, and released.

The compressed sample is removed and sieved in a 3-inch diameter U.S. Standard sieve (W.S. Tyler Company, manufacturer). The fraction retained on the sieve next greater in number (next smaller in screen opening) than the greater number sieve defining the original sample of support is recorded as Percent Survival.

(U) Bulk Density

Packed bulk density, B_D, is determined by weighing a quantity of support or catalyst into a graduated glass cylinder to determine its volume. (In the standard procedure for Shell 405 Catalyst, 200 g of catalyst is weighed into a 250 cc graduate; for this program, because of lesser amounts available, the total amount of support or catalyst prepared was weighed into a 100 cc graduated cylinder.) The bottom of the cylinder is tapped for 3 minutes and the volume of material is then noted. Bulk density is reported on a dry basis, i.e., corrected for loss on ignition (L.O.I.) and calculated as follows:

$$B_D \text{ (dry basis)} = \frac{\text{Weight} - \text{Weight (L.O.I./100)}}{\text{Volume}}$$

(L.O.L., %w, is determined by heating a weighted (1-2 g) sample of support in a small porcelain crucible at approximately 700 C for 30 minutes, cooling in a dissicator, and reweighing).

APPENDIX III

Graphs of BET Nitrogen Adsorption Data Output-Pore Diameter as a Function of Specific Pore Volume Per Unit Diameter

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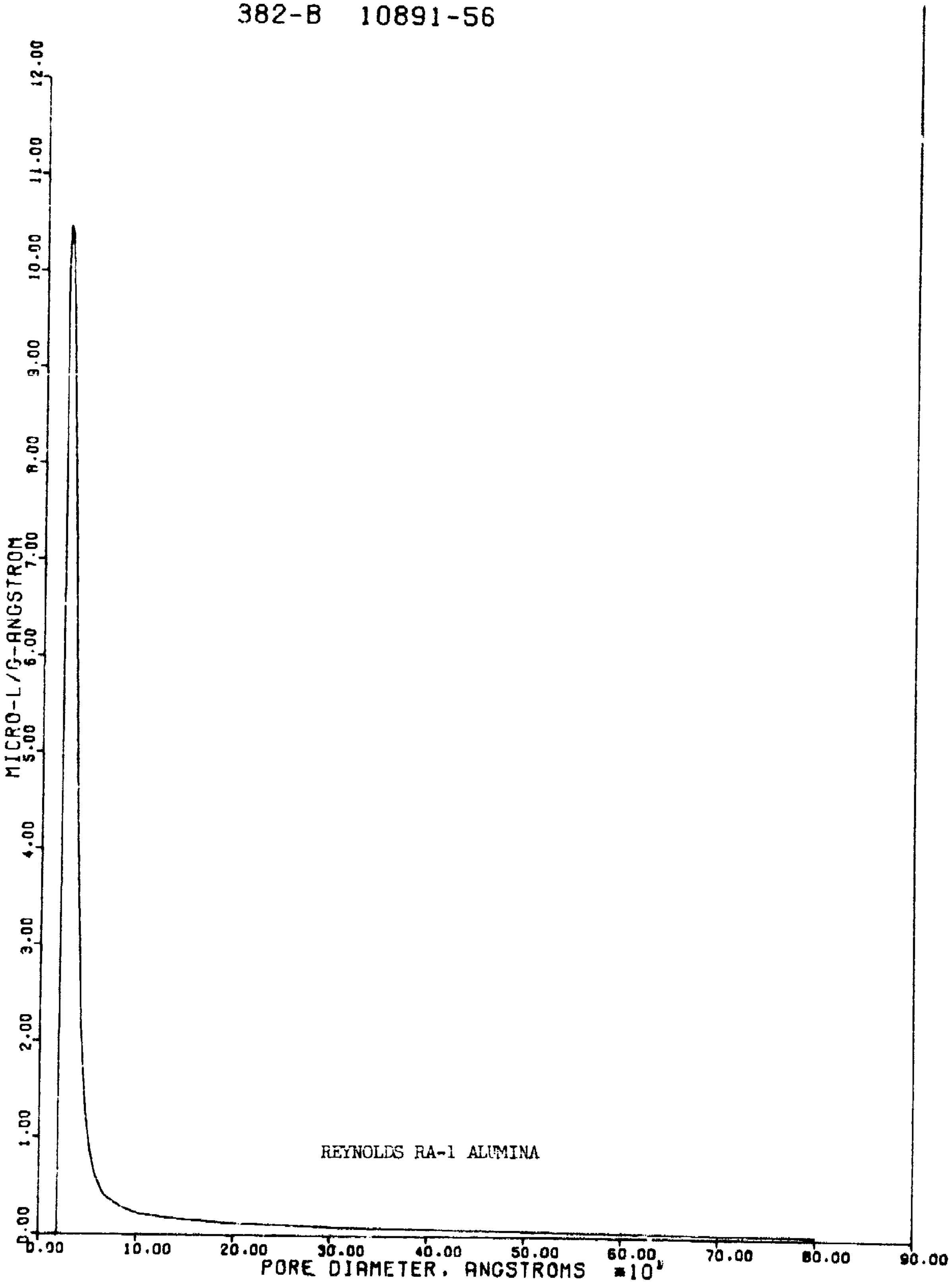
382-B 10891-56



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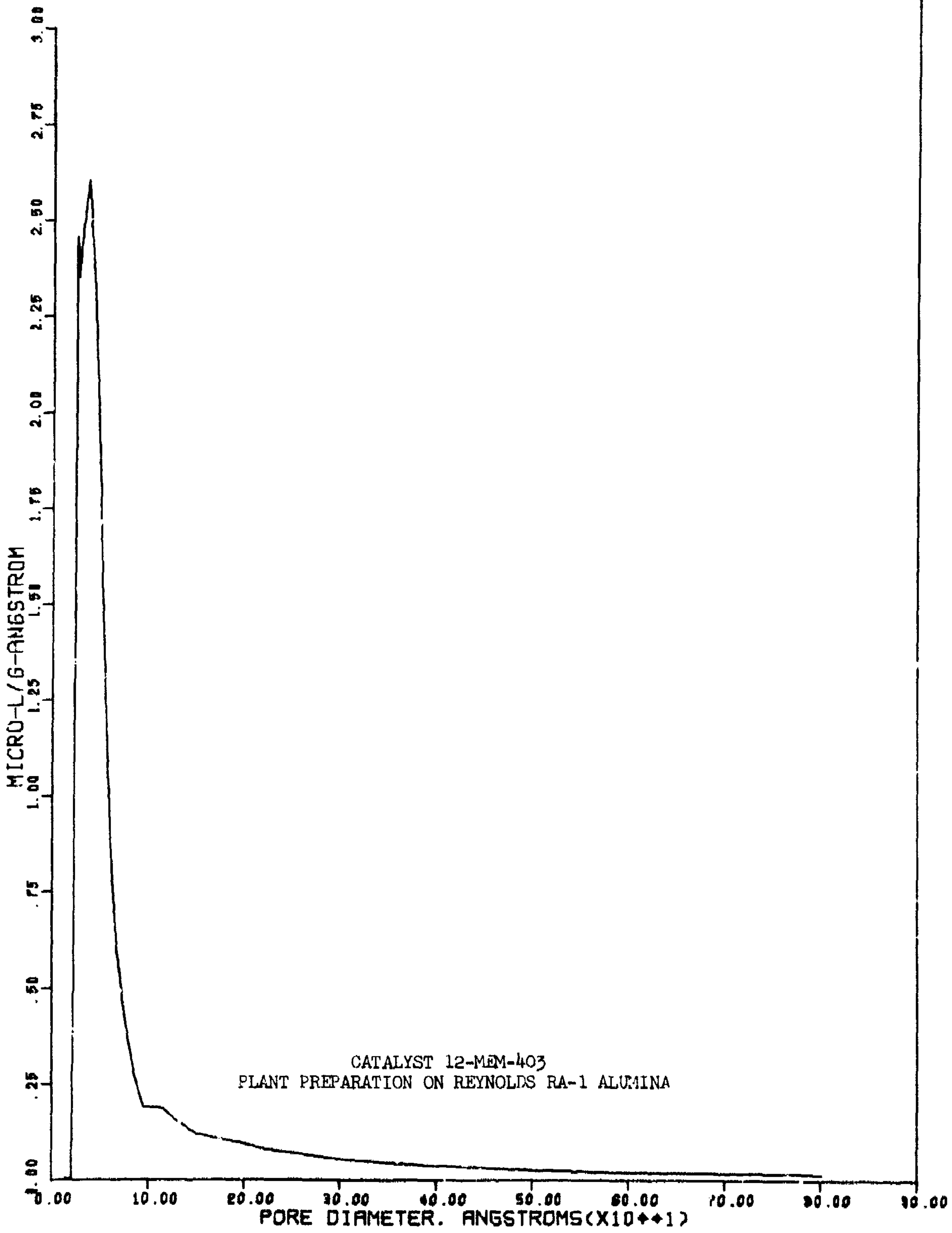

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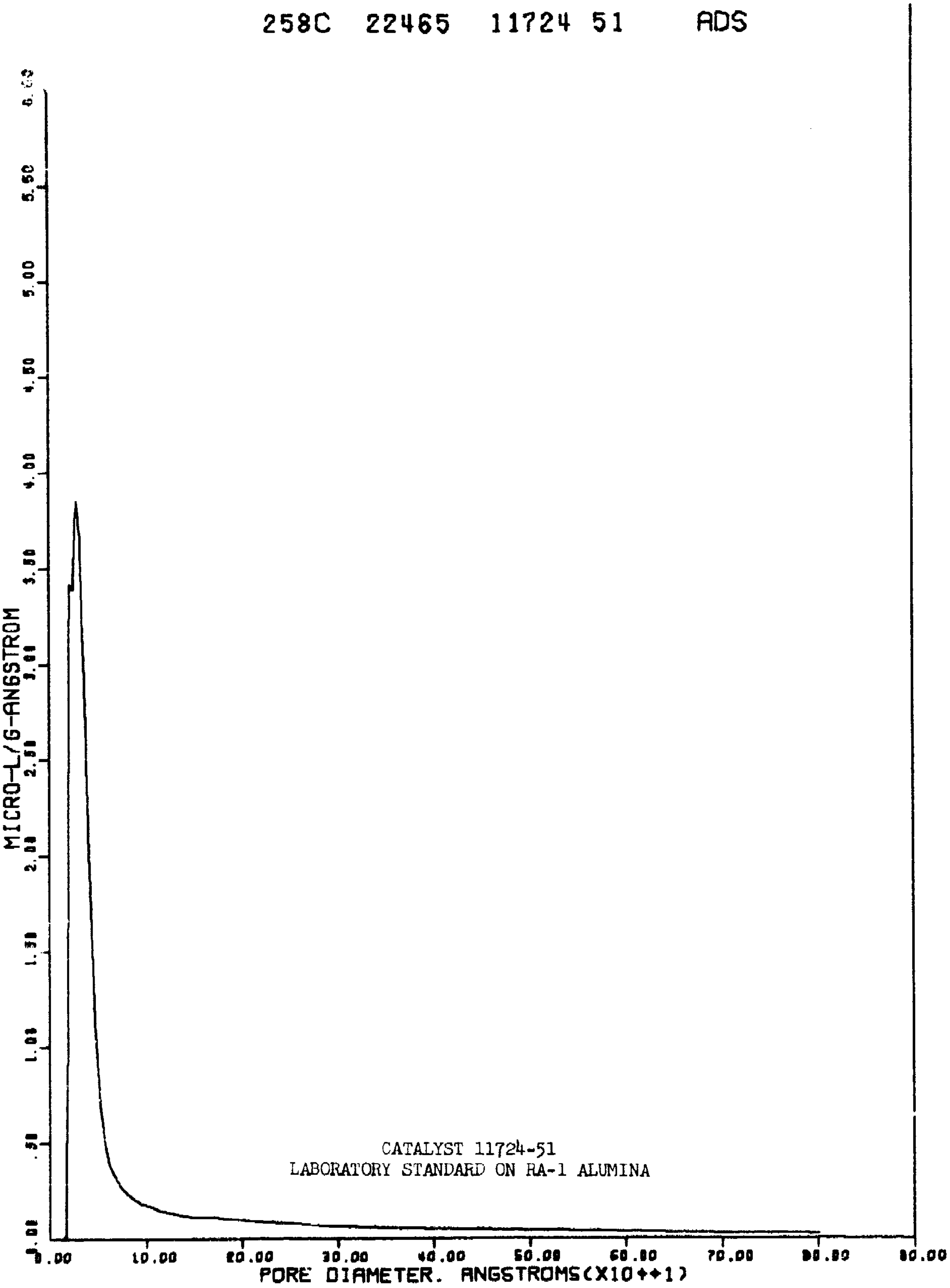

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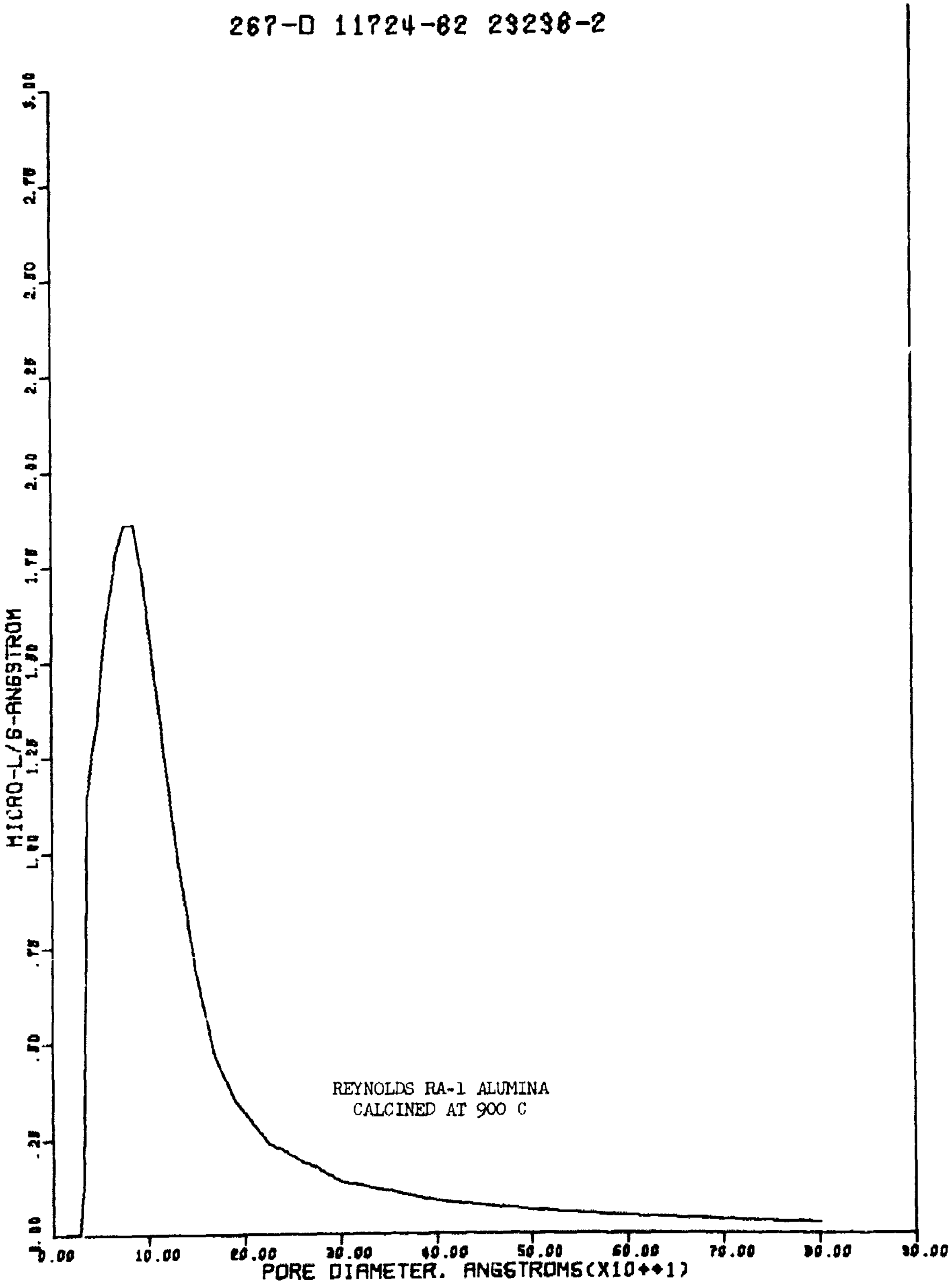

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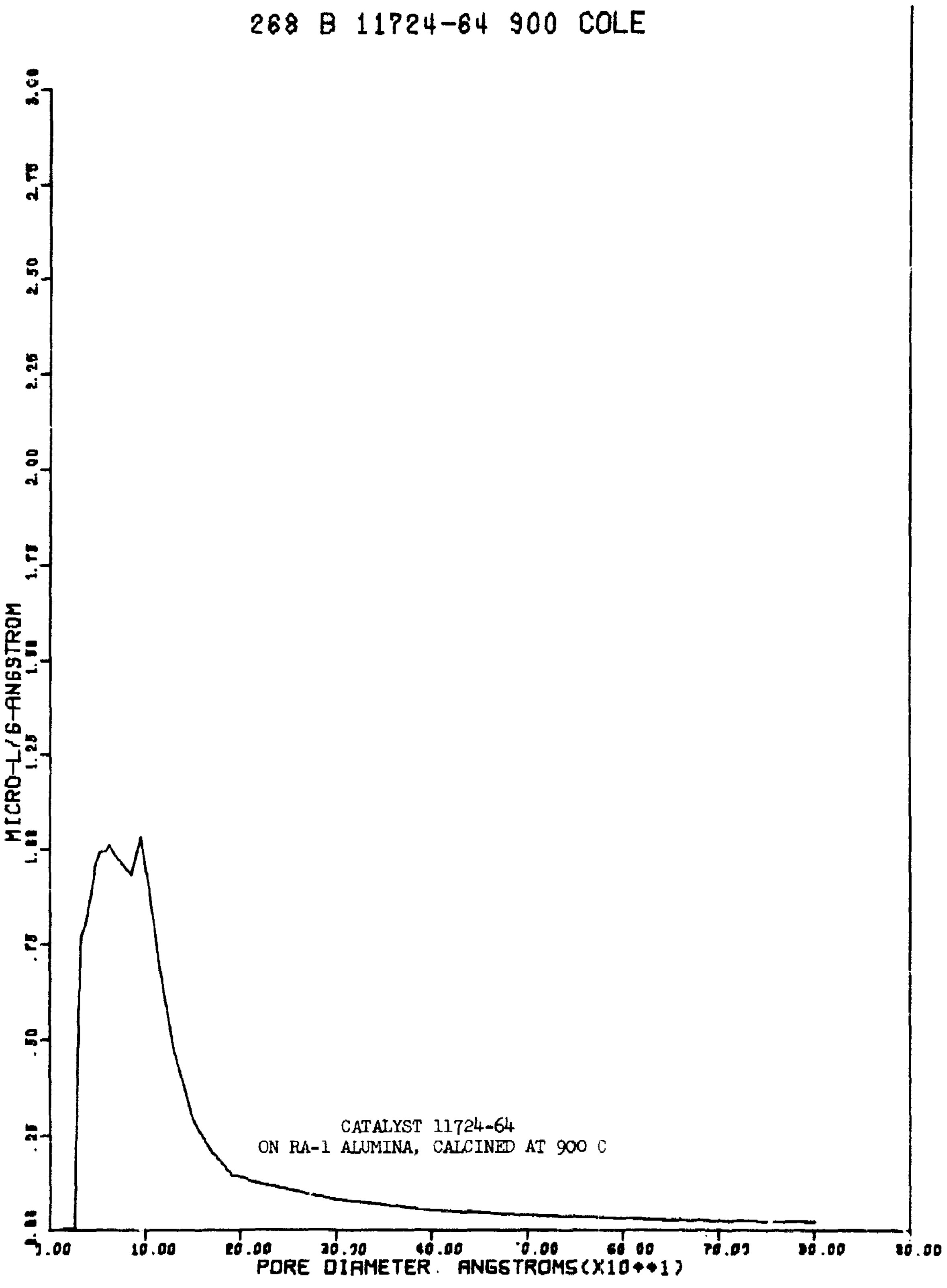
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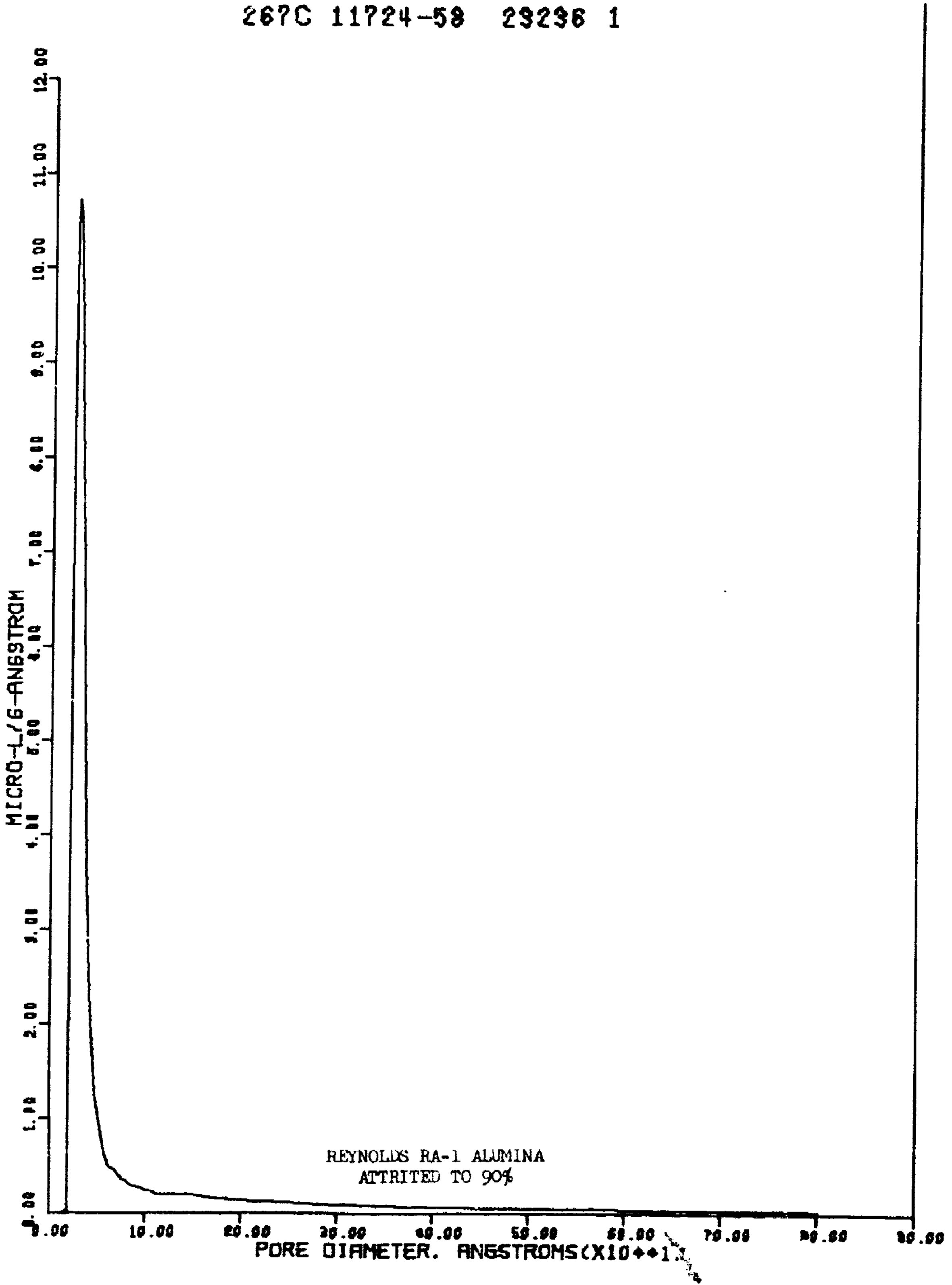
6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ATTRITED TO 90%
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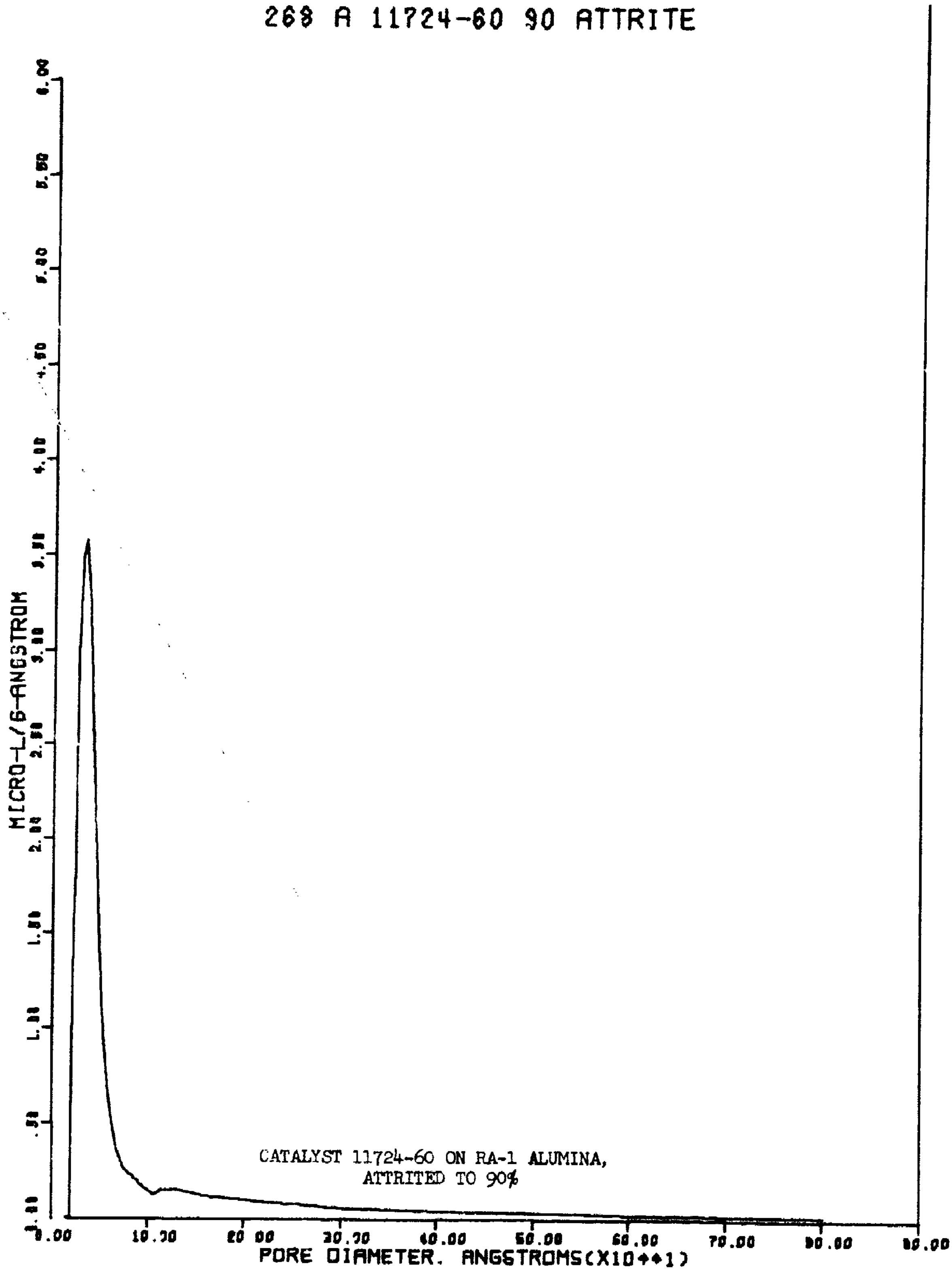

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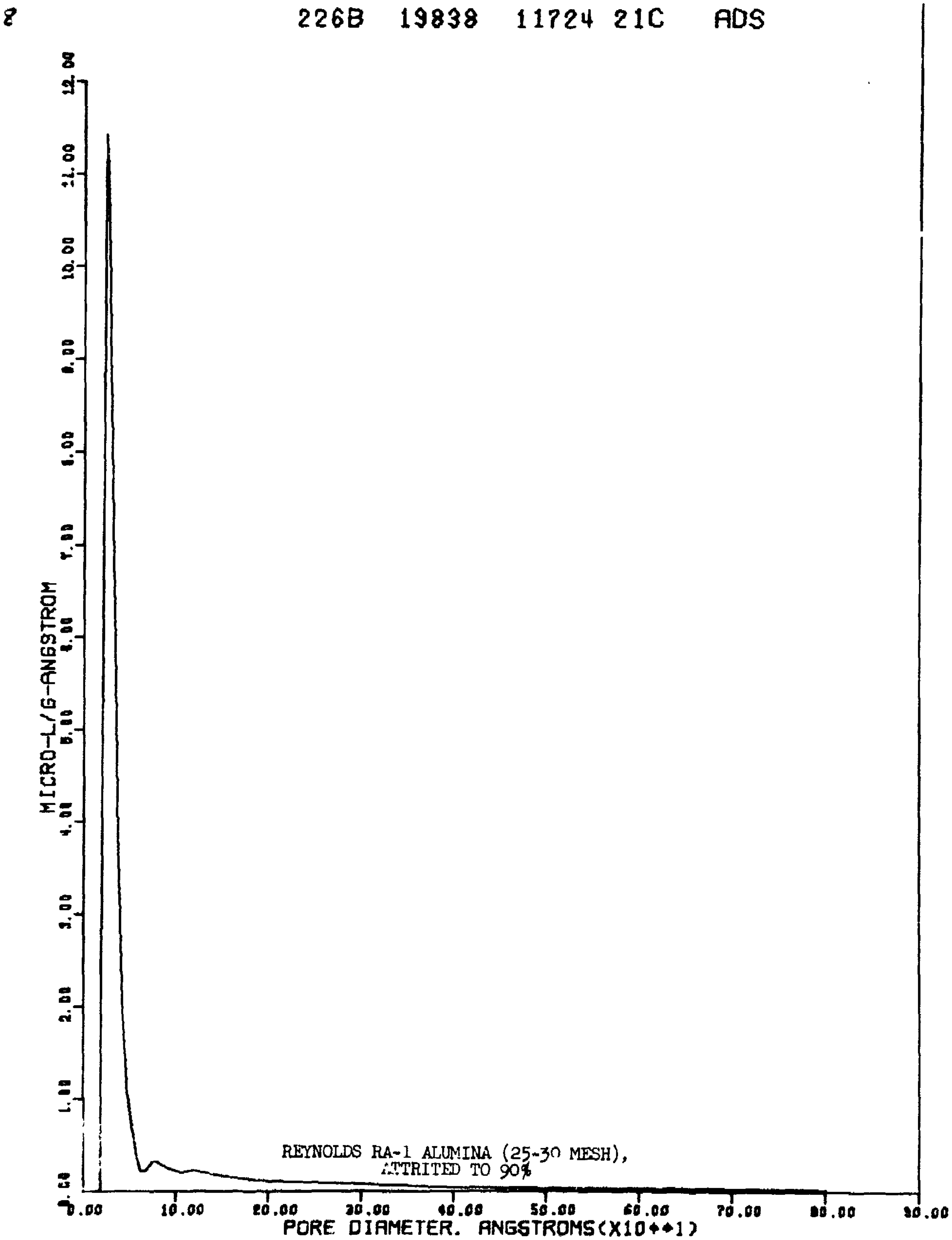
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PORE DIAMETER. ANGSTROMS(X10**1)
REYNOLDS RA-1 ALUMINA (25-30 MESH),
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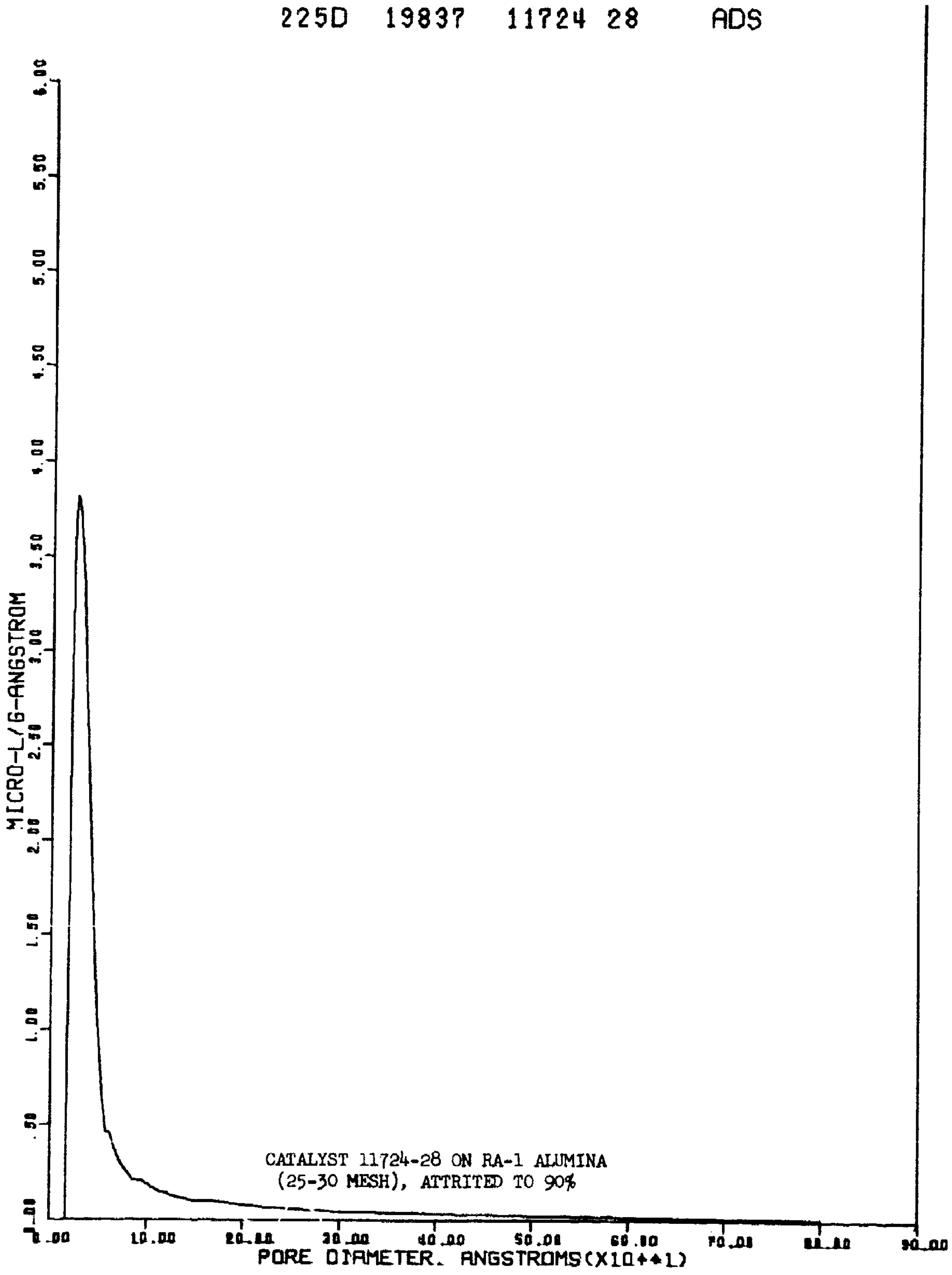

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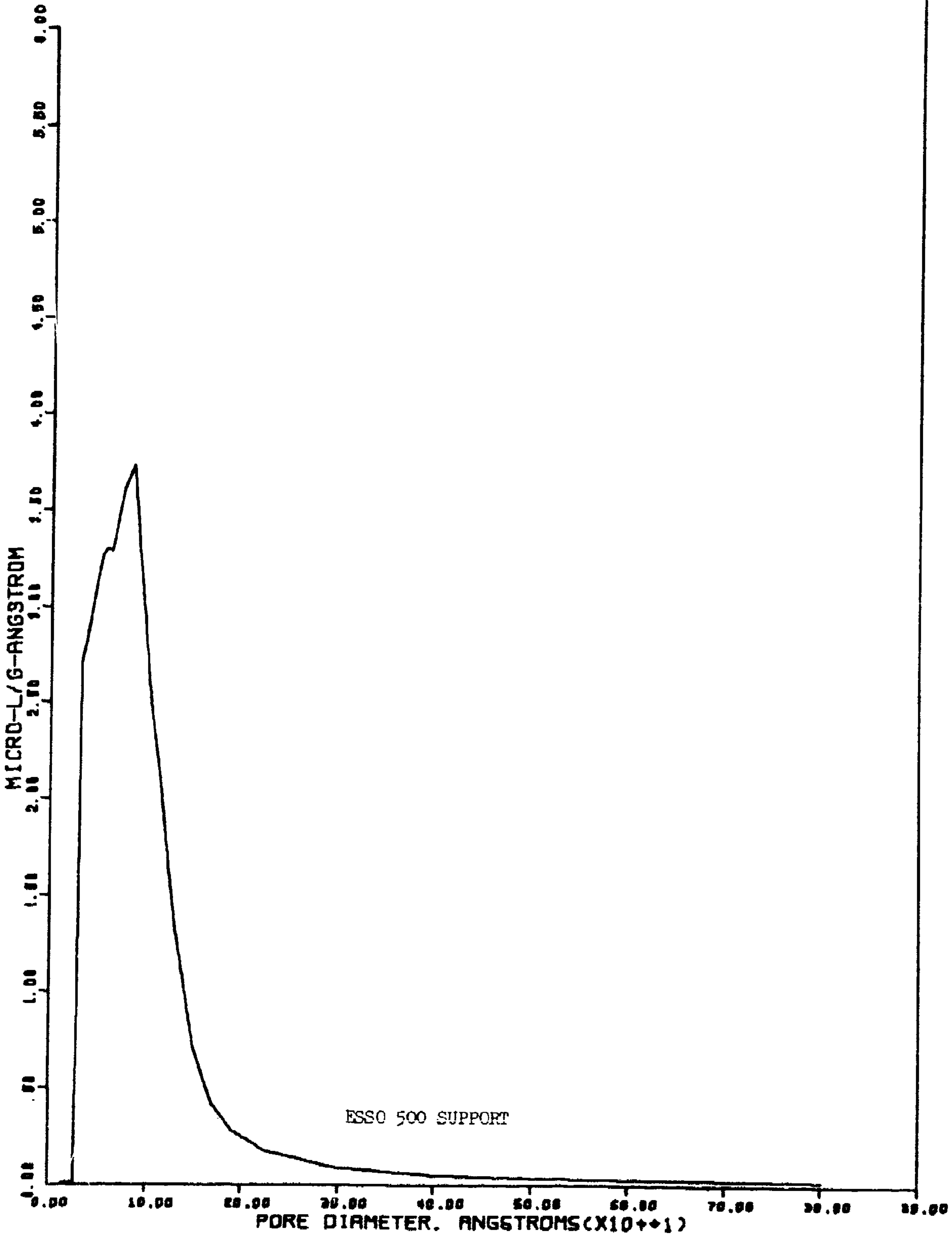

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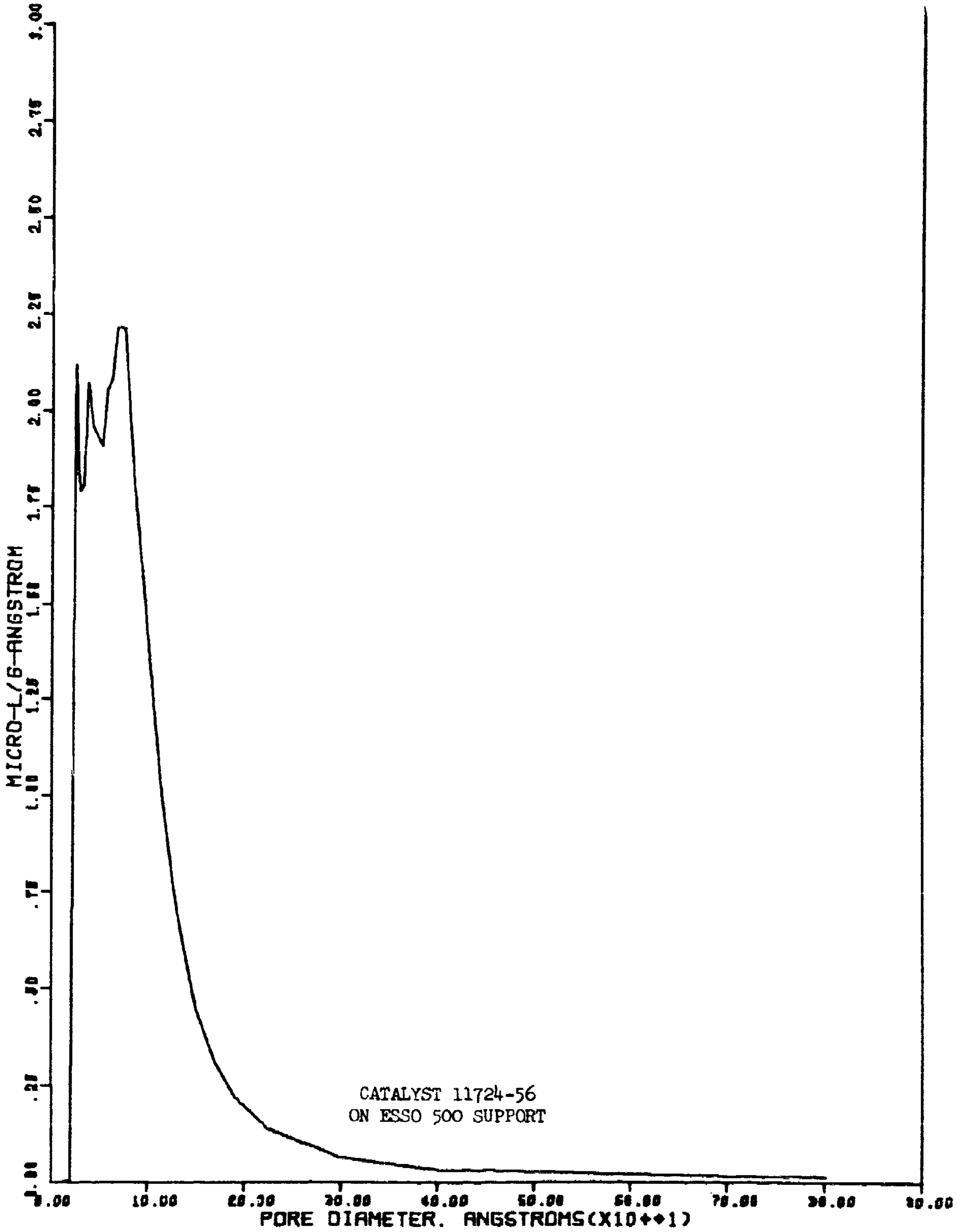

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ON ESSO 500 SUPPORT

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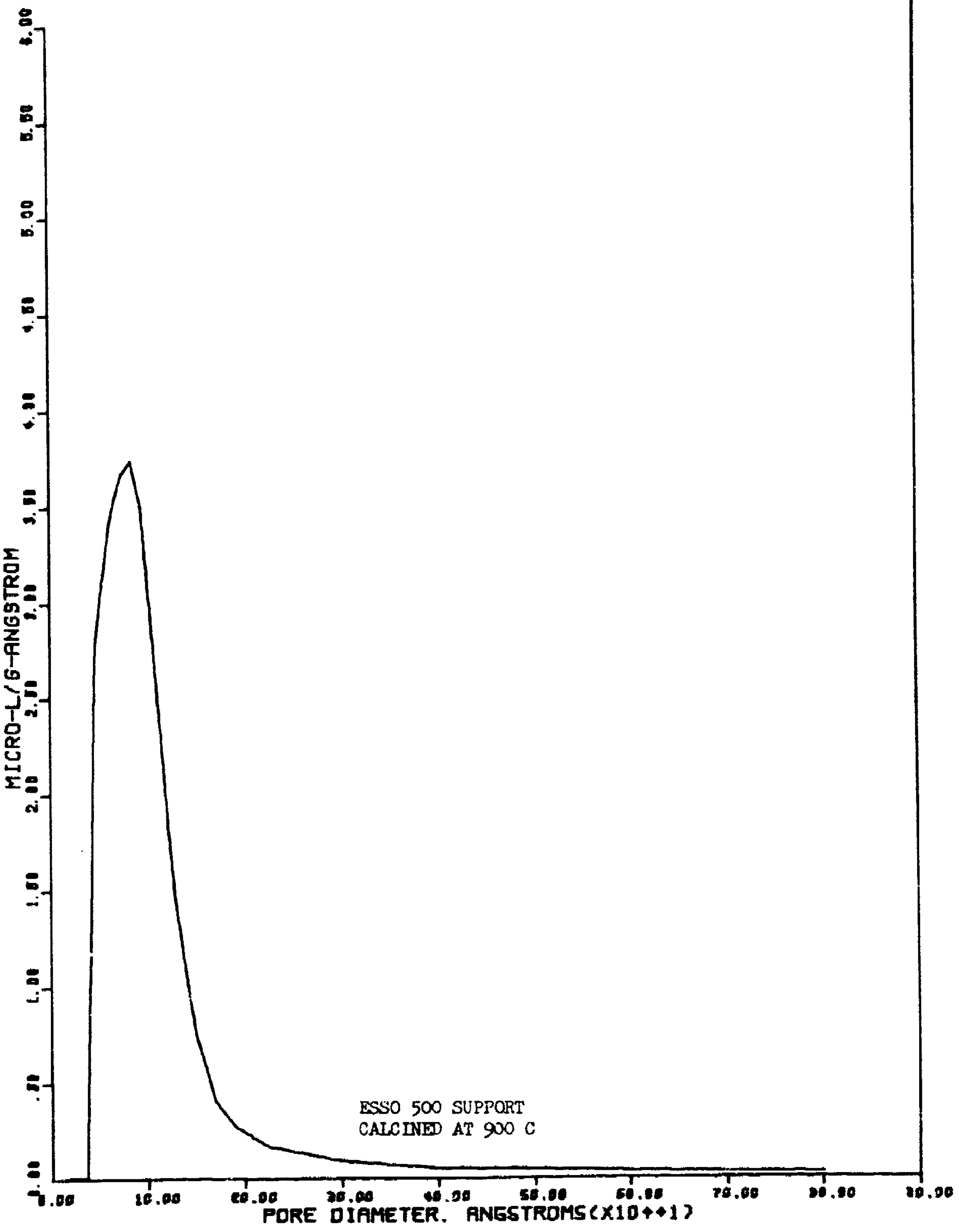

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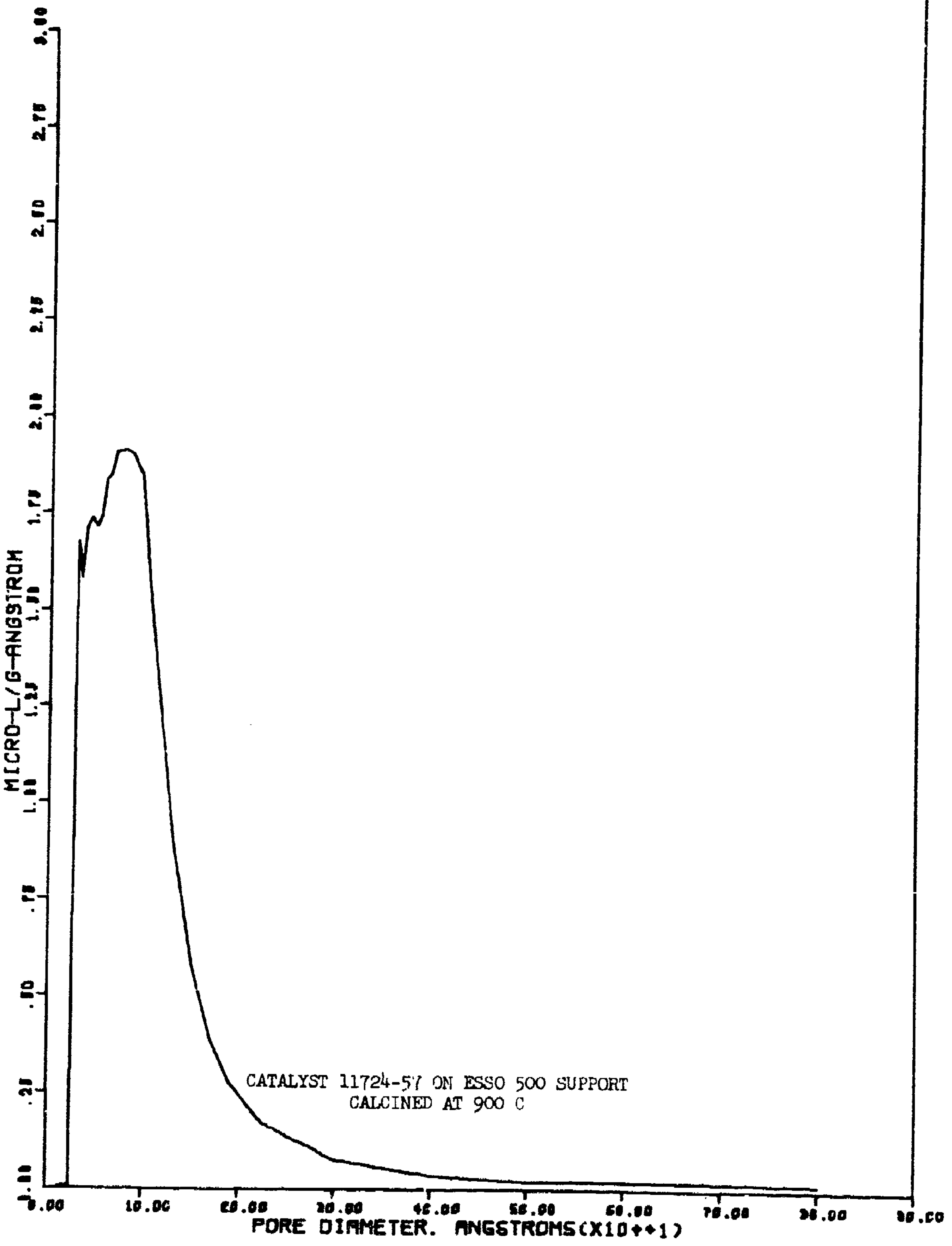

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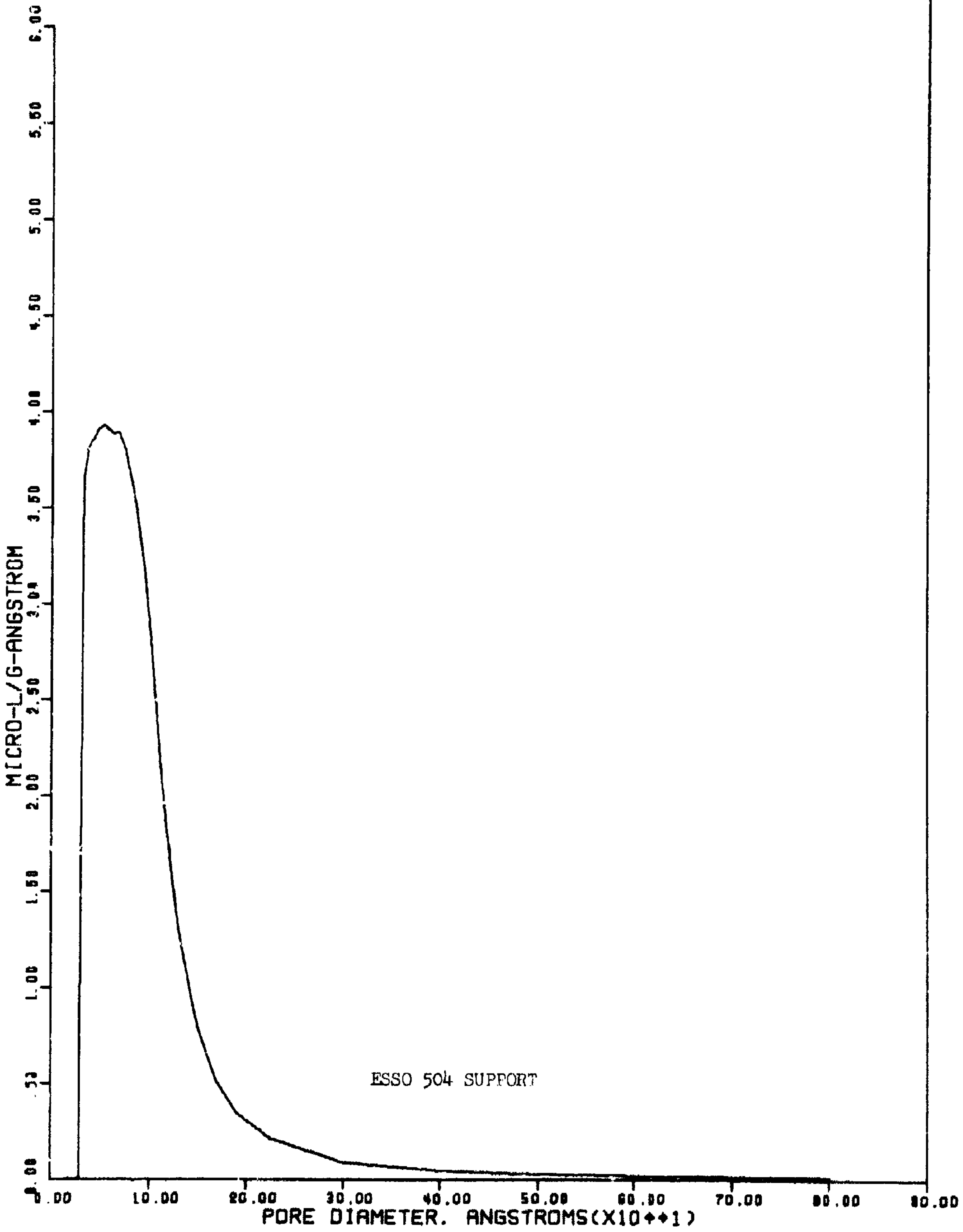

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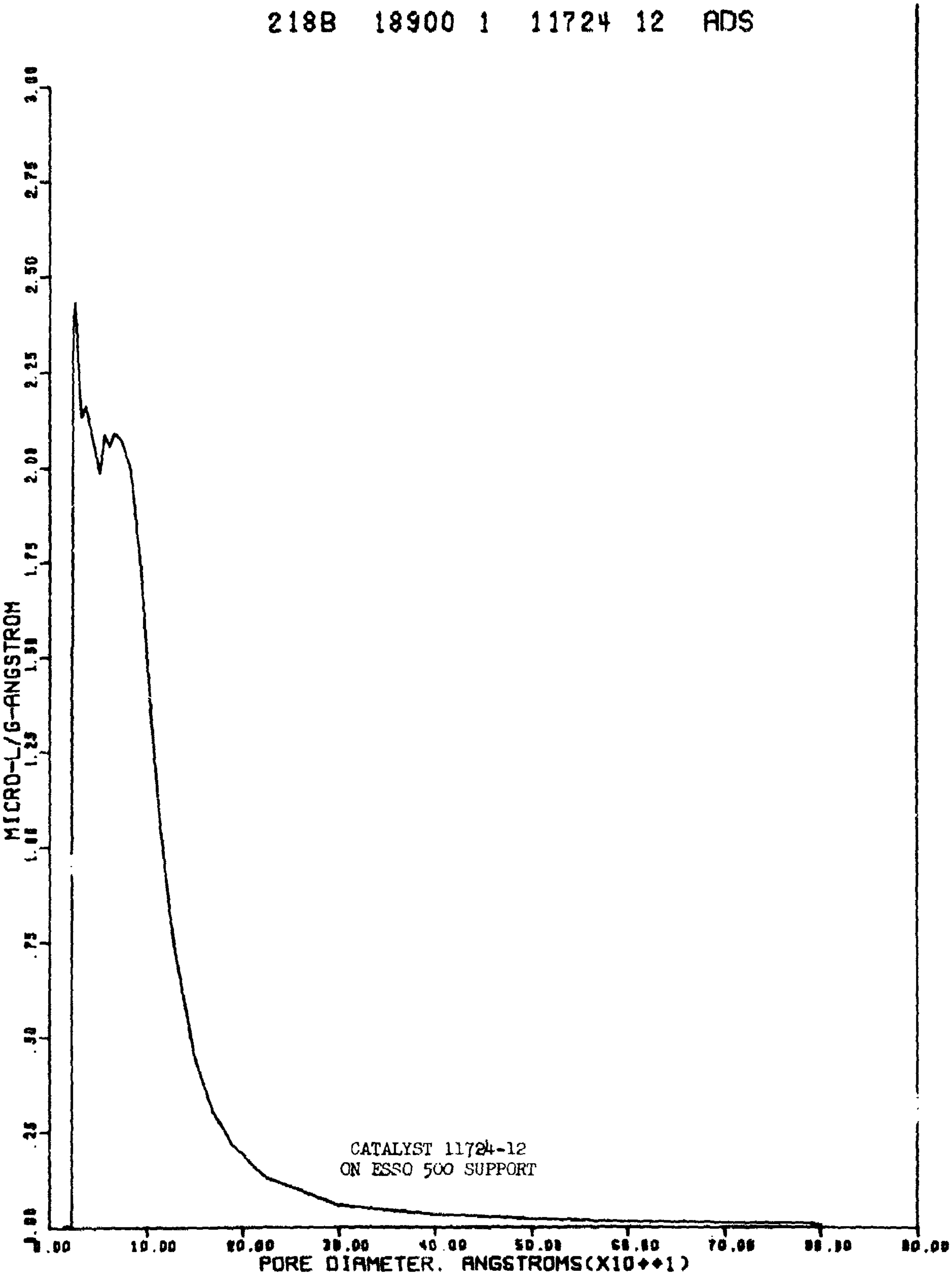

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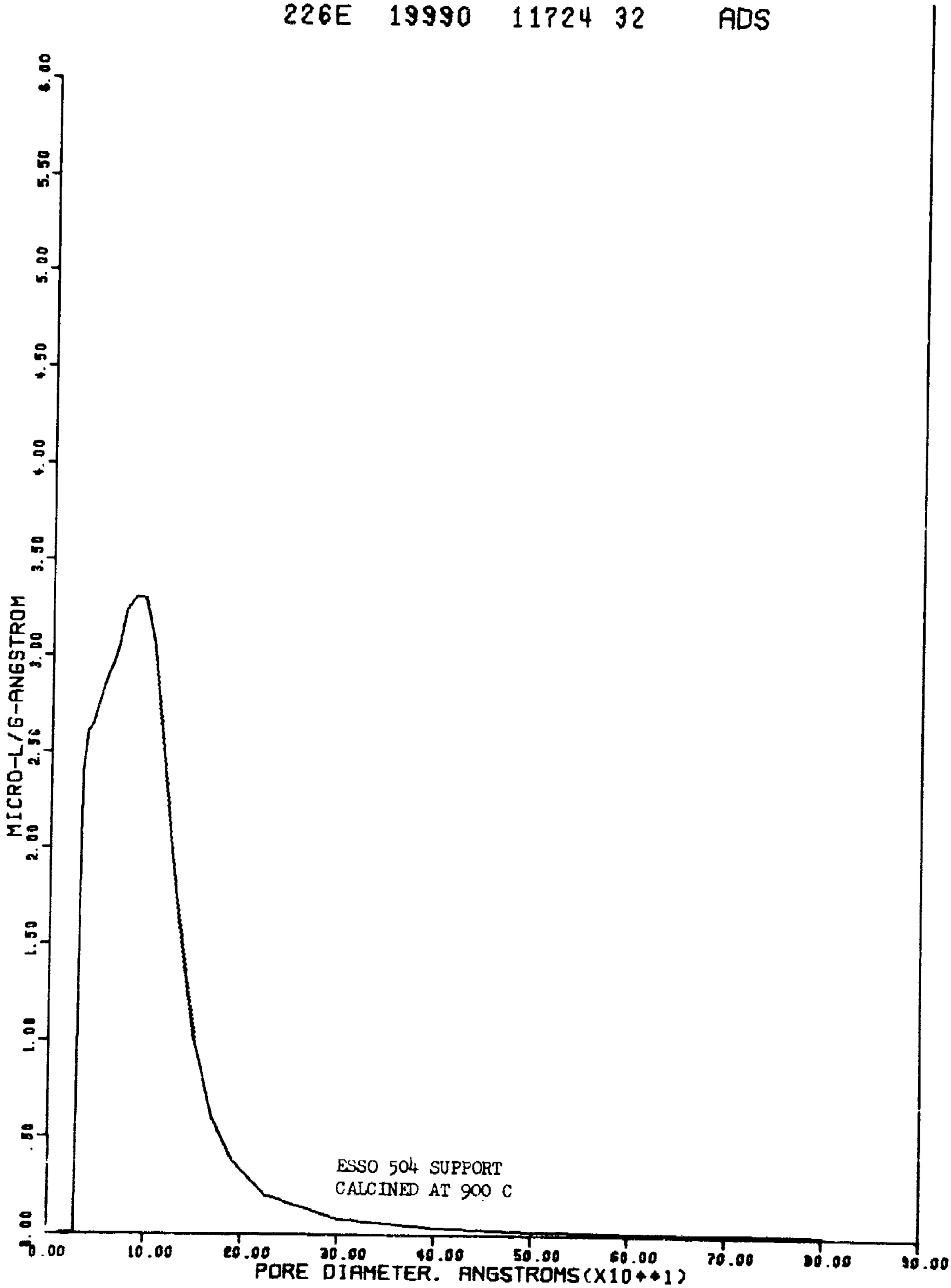

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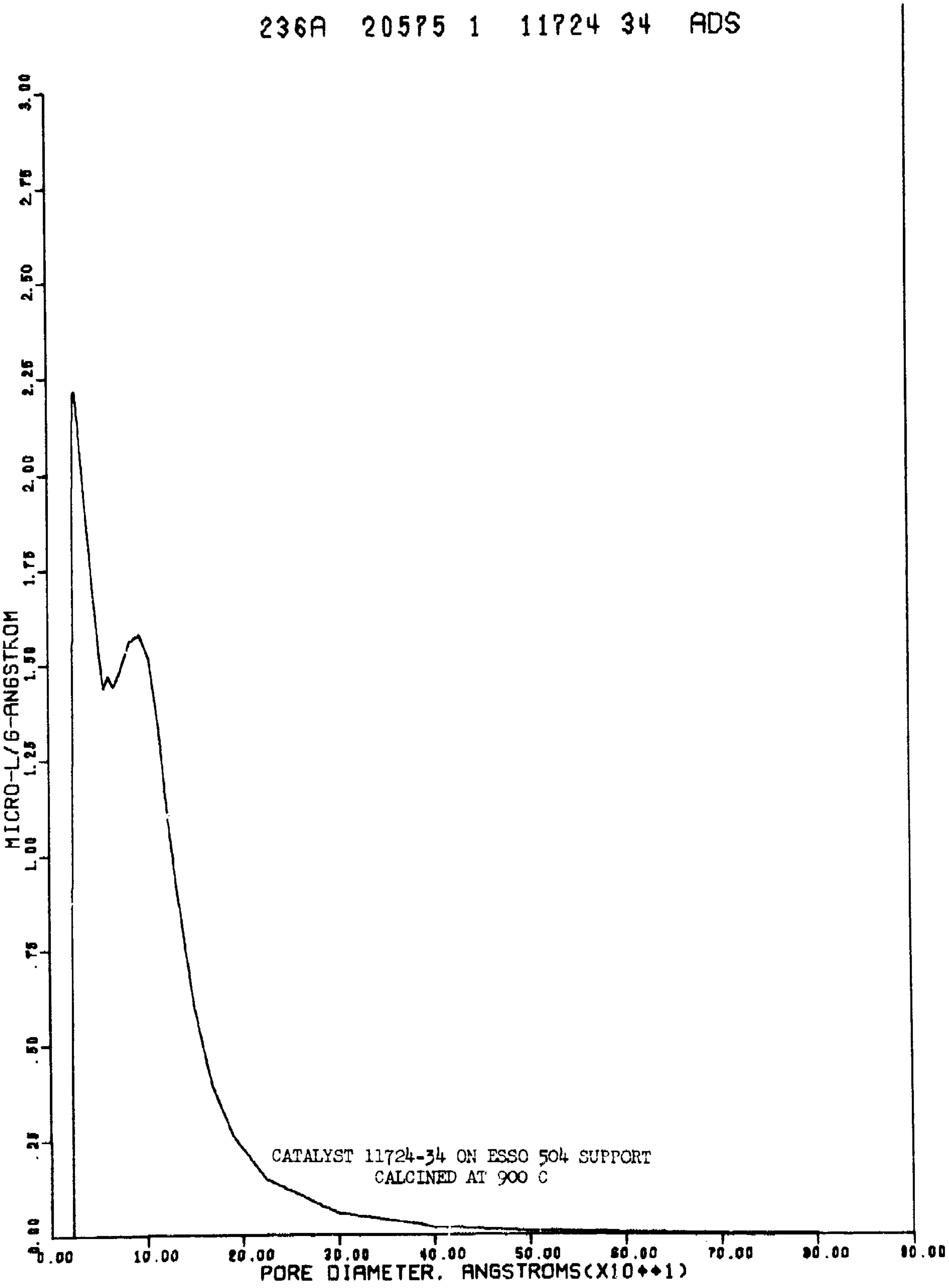

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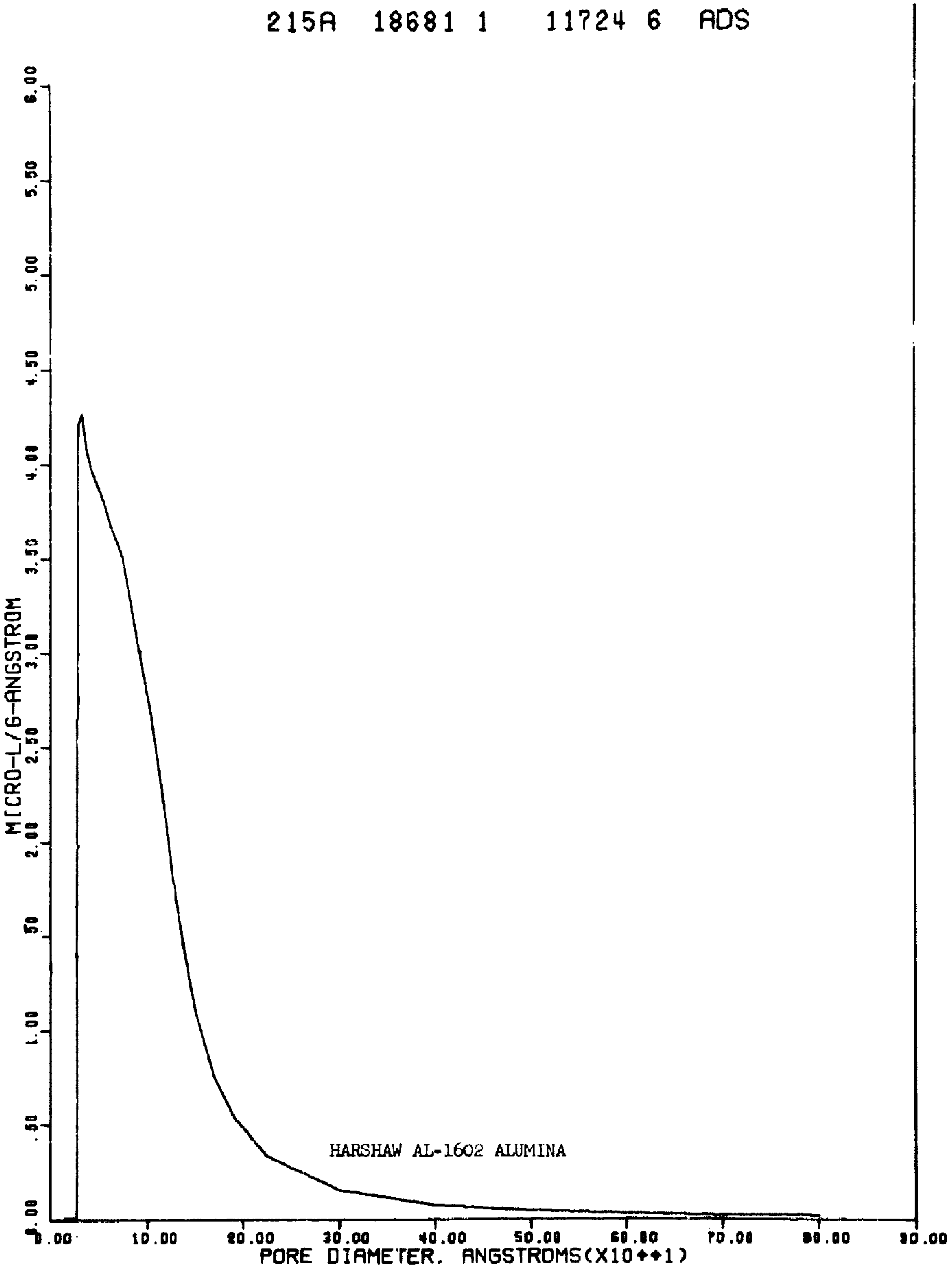
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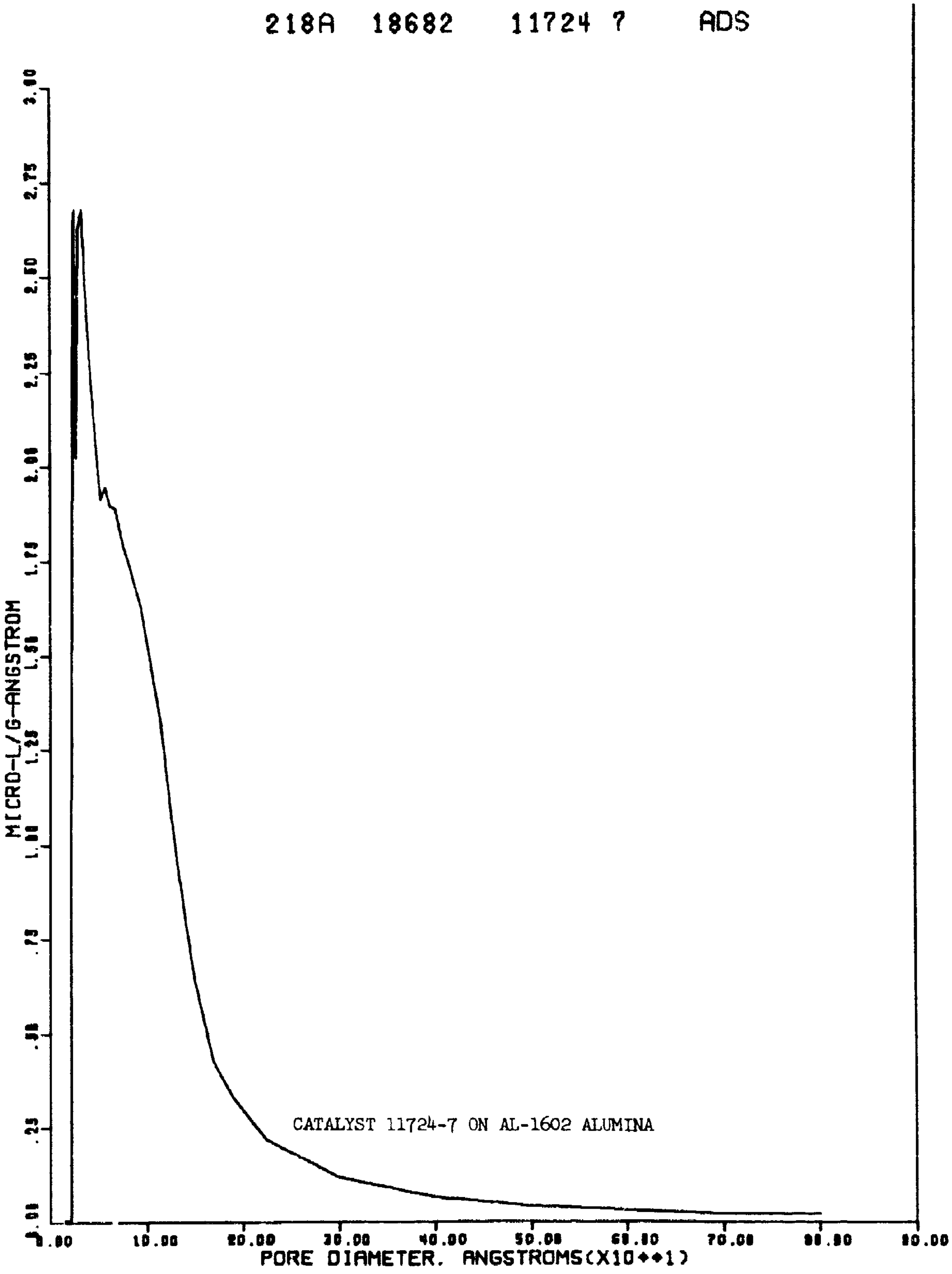

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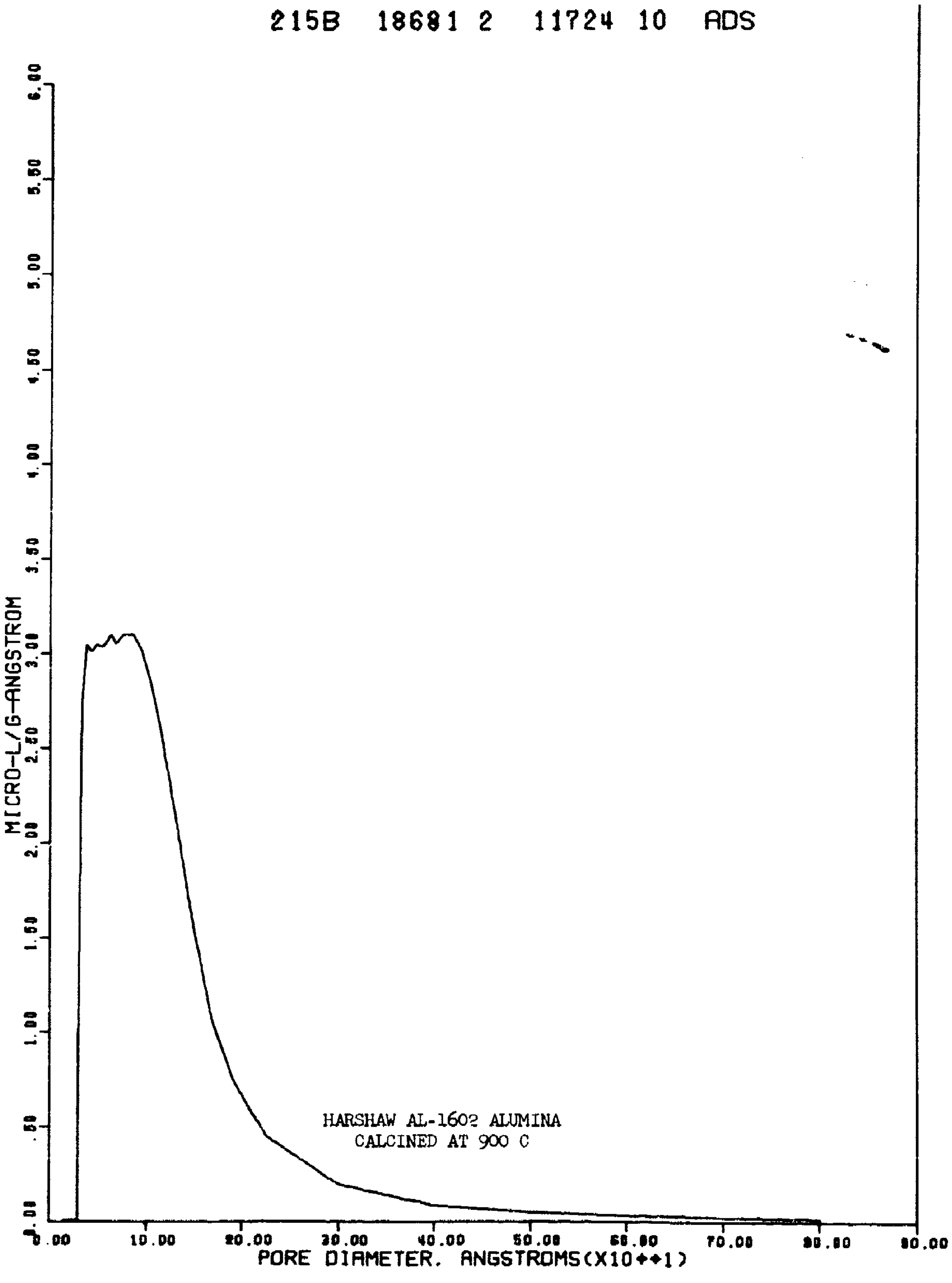
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CALCINED AT 900 C

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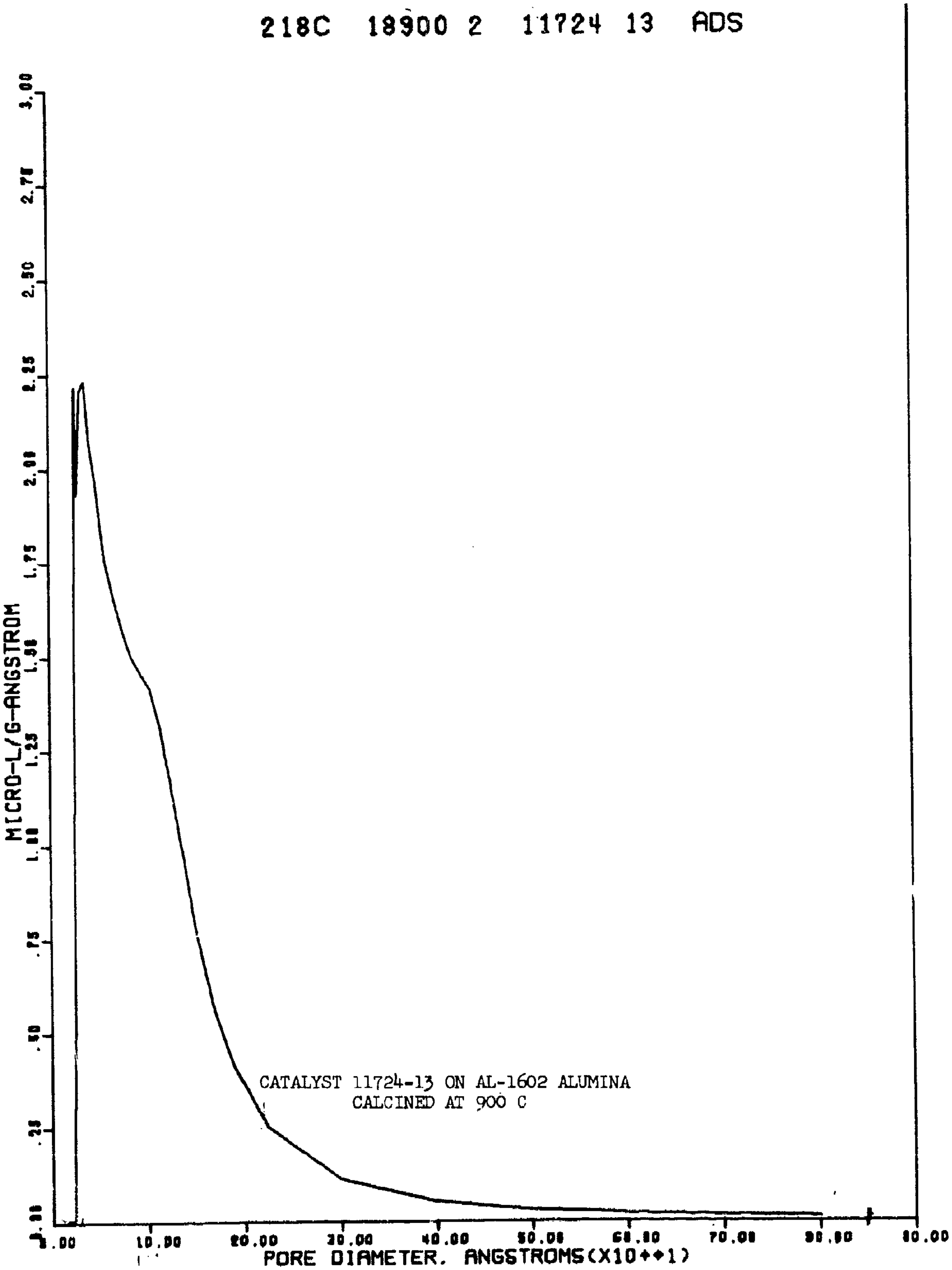

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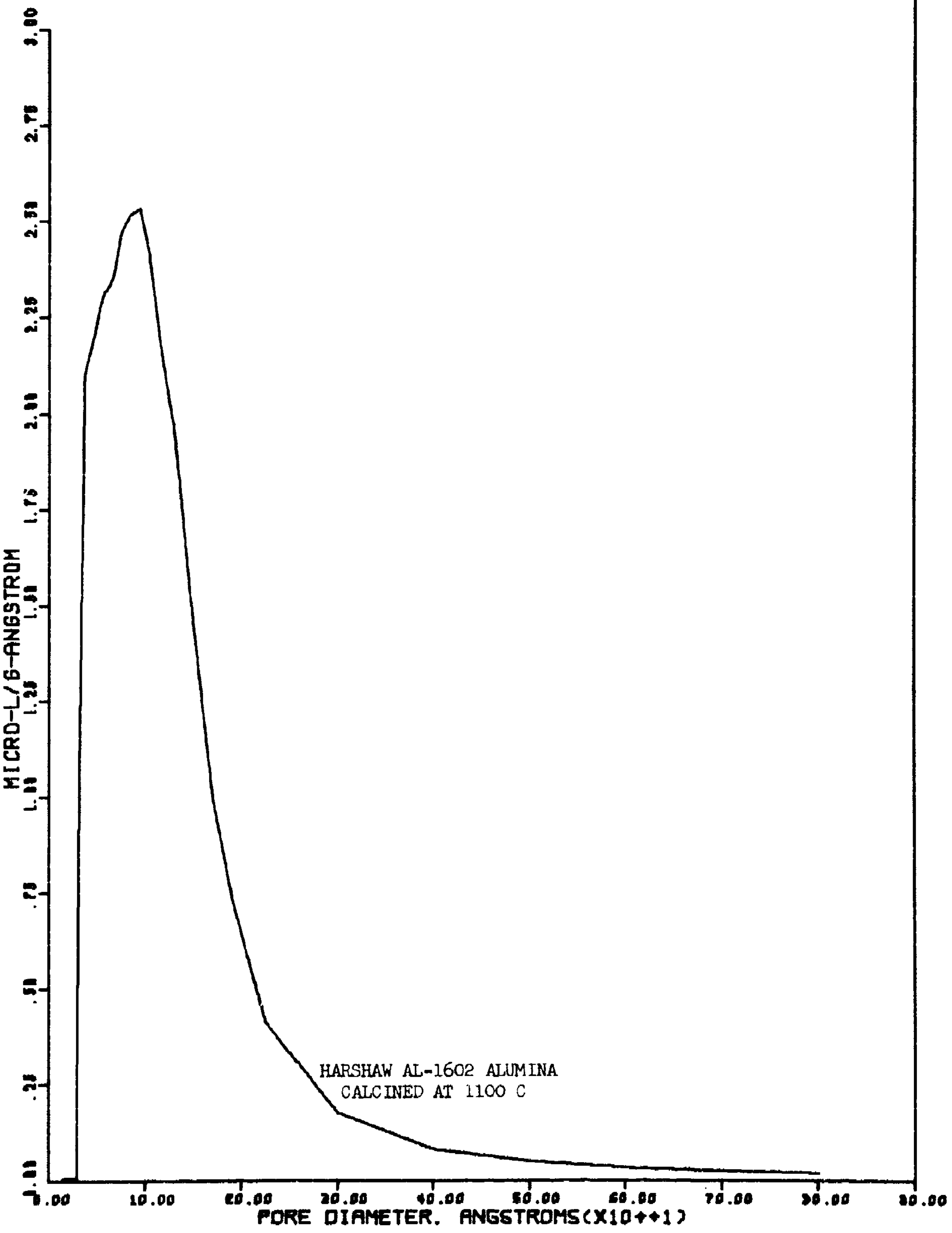
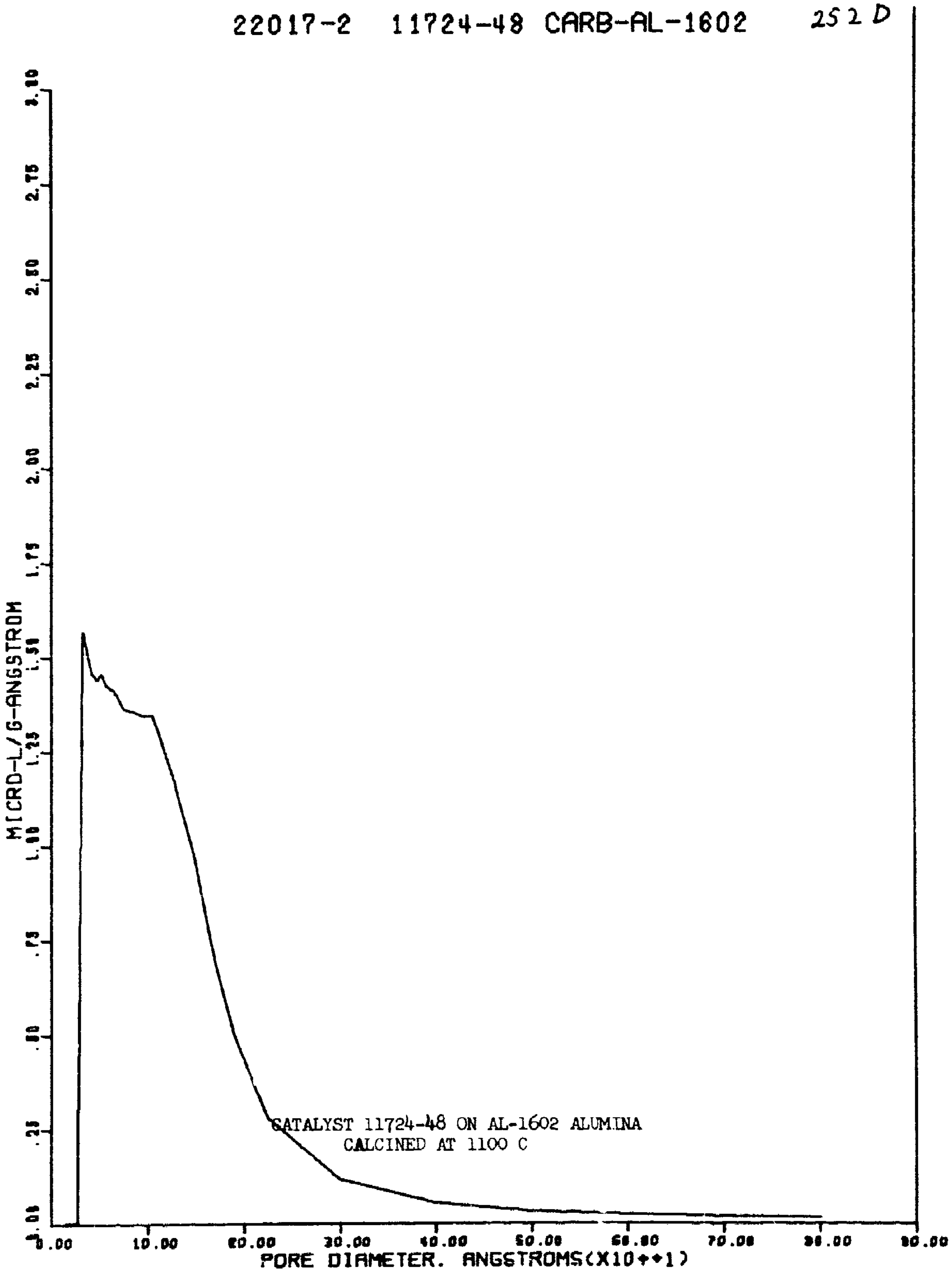


HARSHAW AL-1602 ALUMINA
CALCINED AT 1100 C

23

252 D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CATALYST 11724-48 ON AL-1602 ALUMINA
CALCINED AT 1100 C

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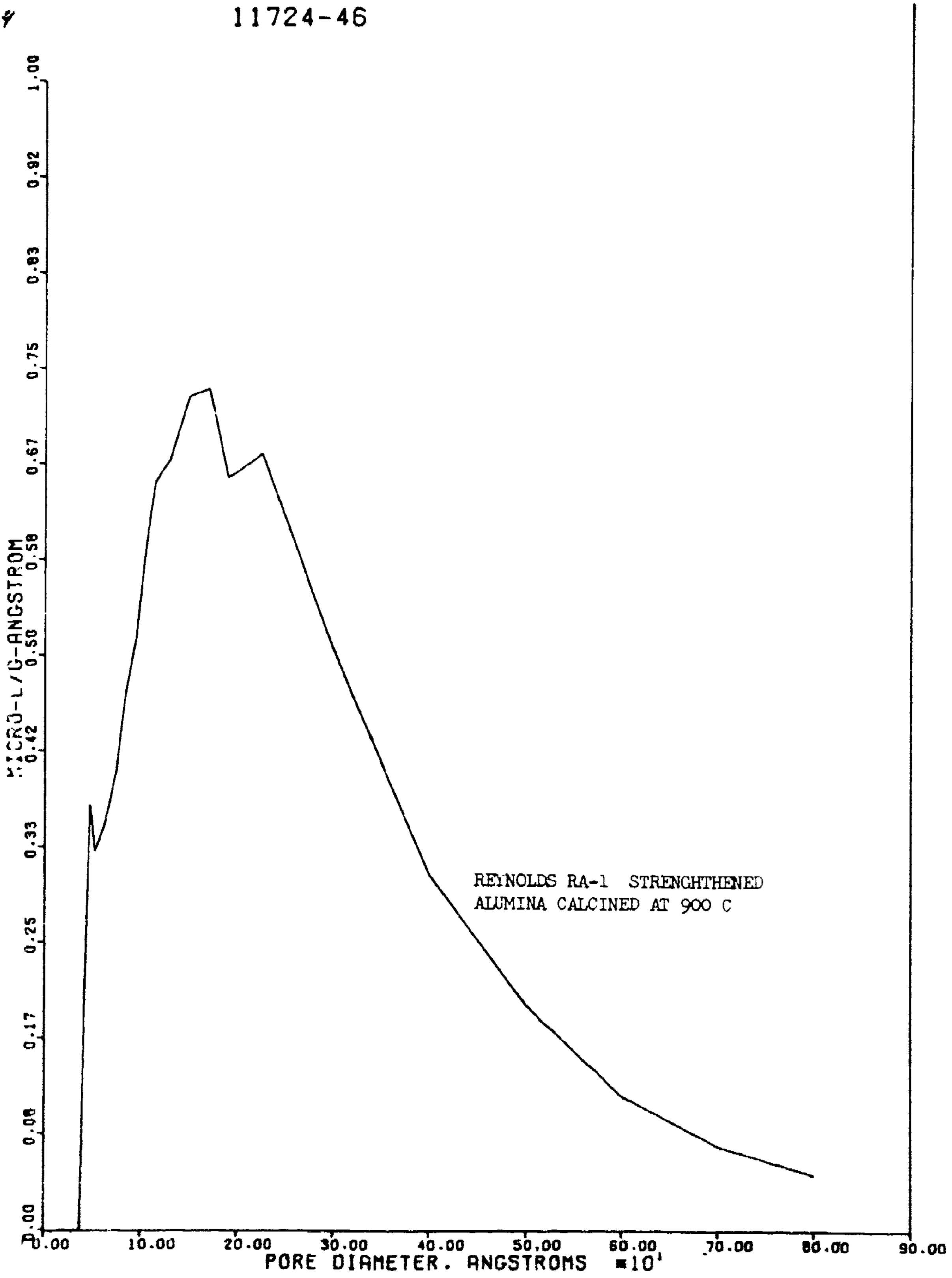
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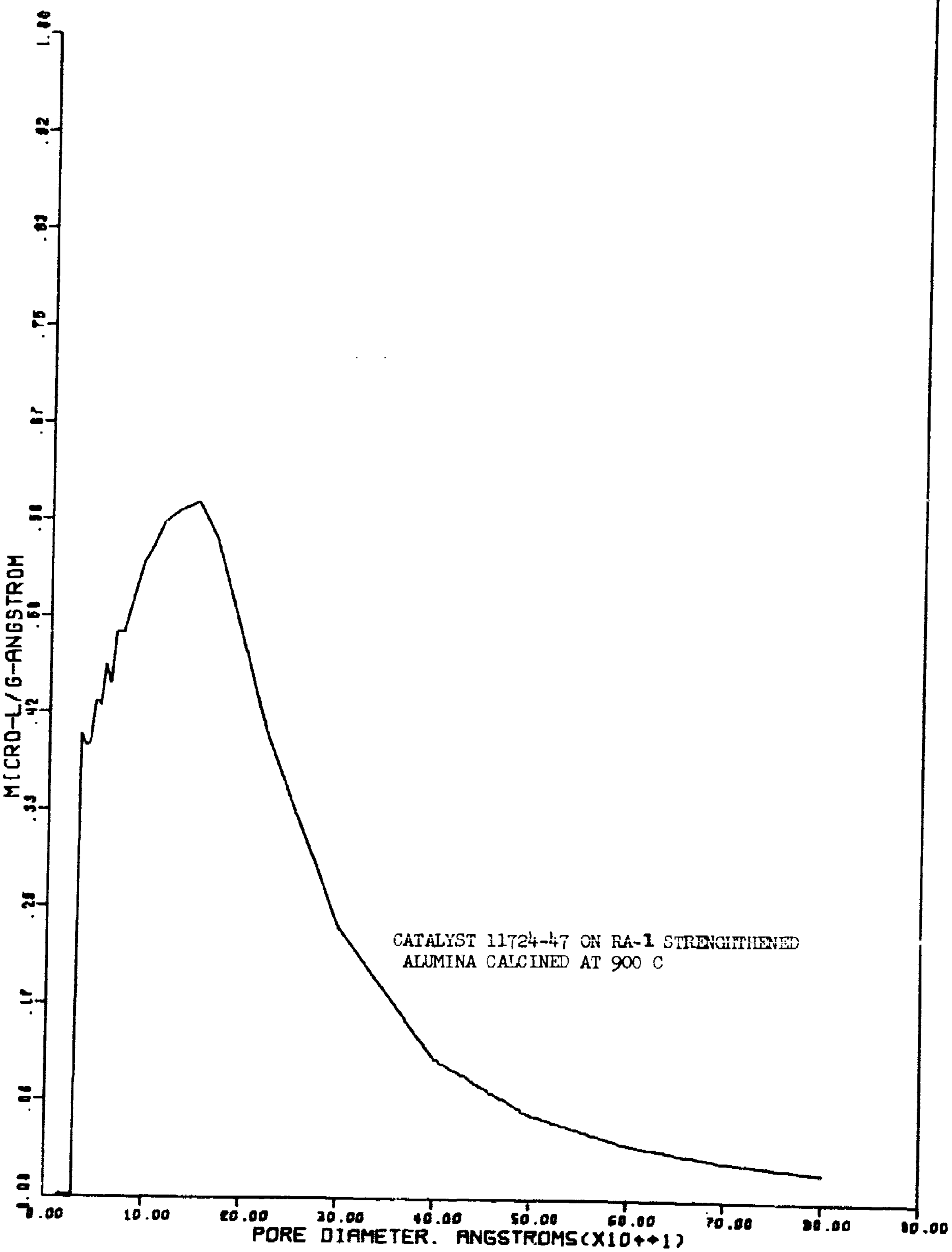
20.00
30.00
40.00
50.00
60.00
70.00
80.00
90.00
PORE DIAMETER, ANGSTROMS $*10^1$
REYNOLDS RA-1 STRENGHTHENED
ALUMINA CALCINED AT 900 C

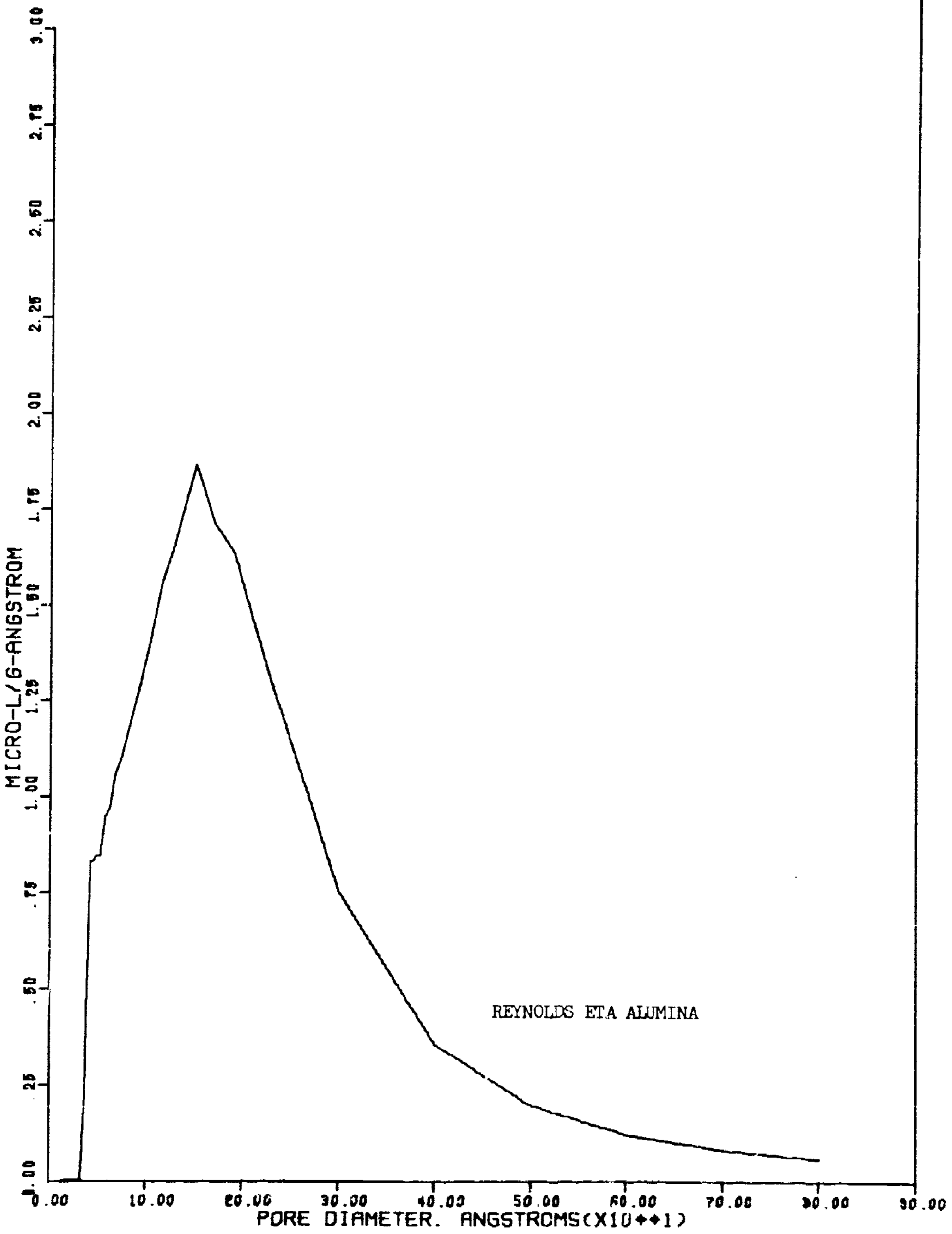
22017-1 11724-47 EXP-RA-1 25 2 C



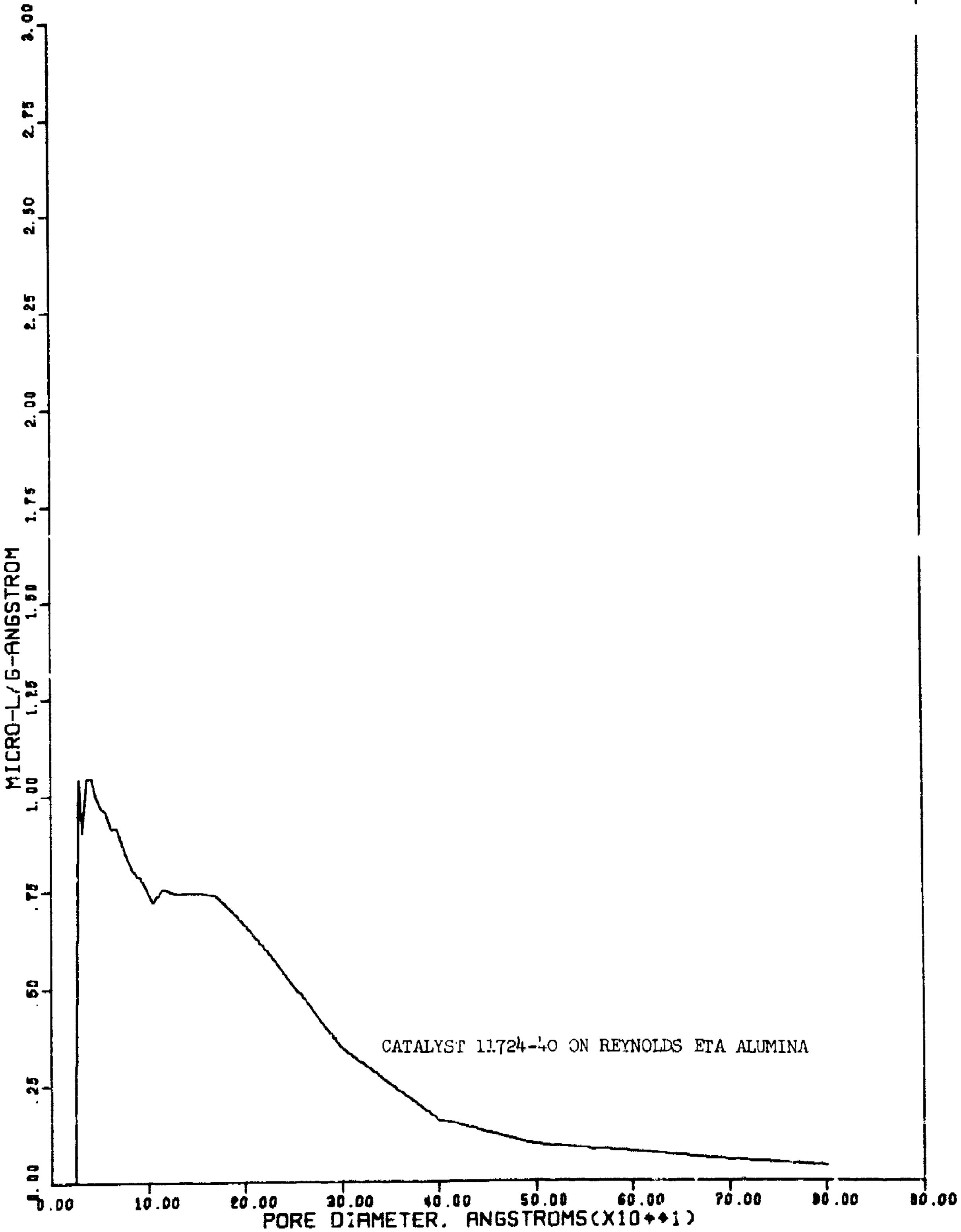
CATALYST 11724-47 ON RA-1 STRENGHTHENED
ALUMINA CALCINED AT 900 C

26

290-A 25925-1 11724-39

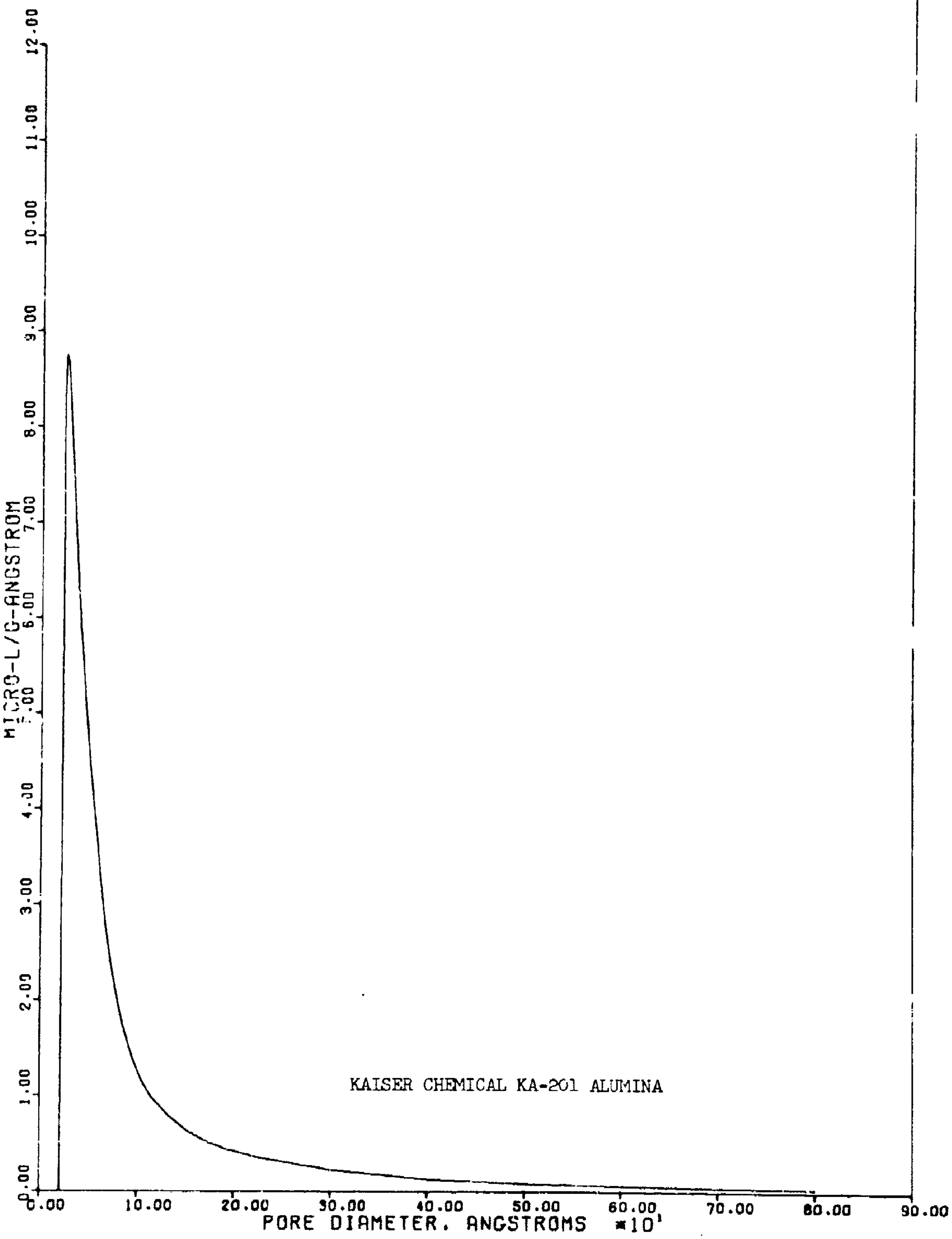


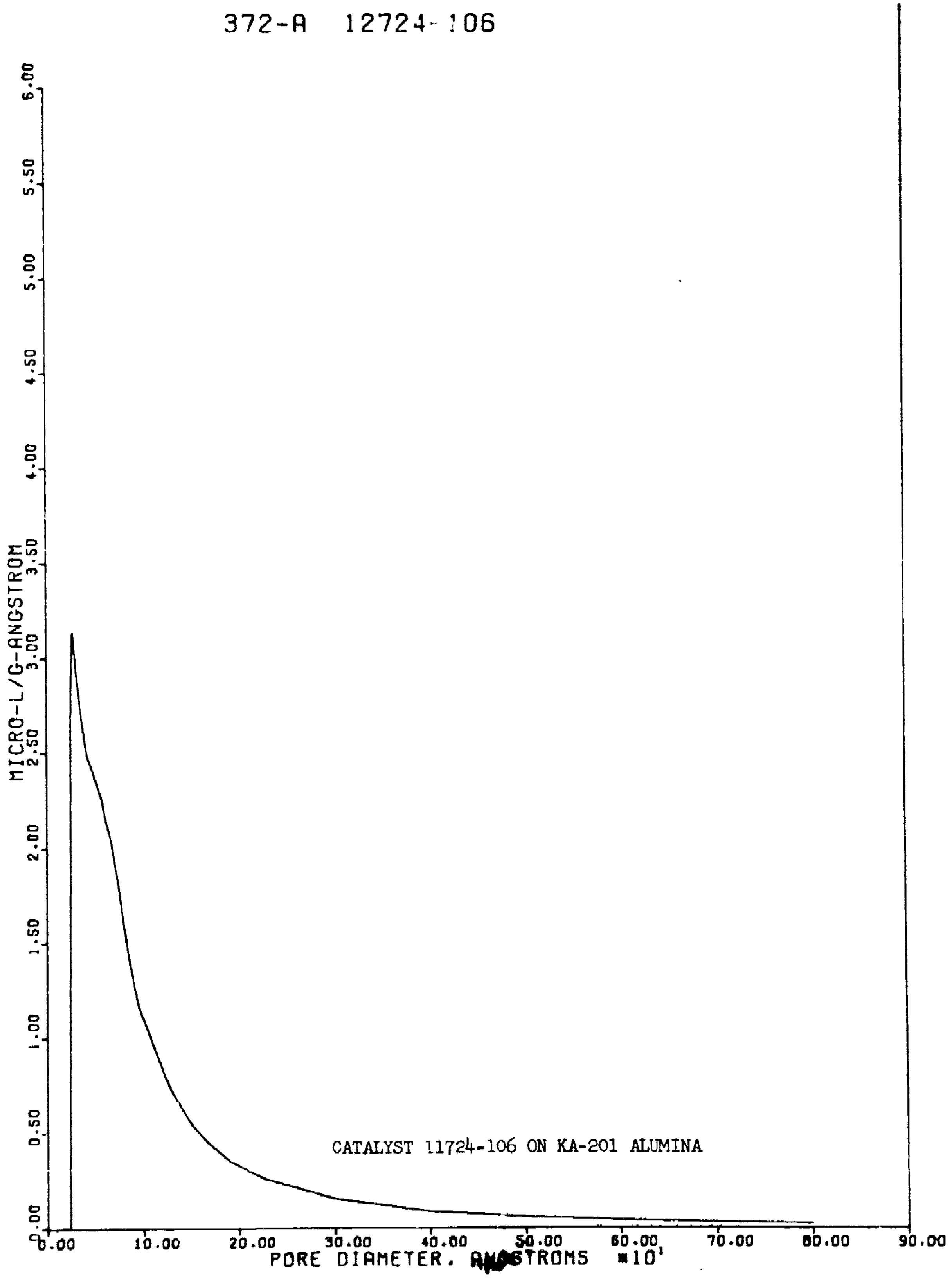
236B 20575 2 11724 40 ADS



28

372-E 11724-104

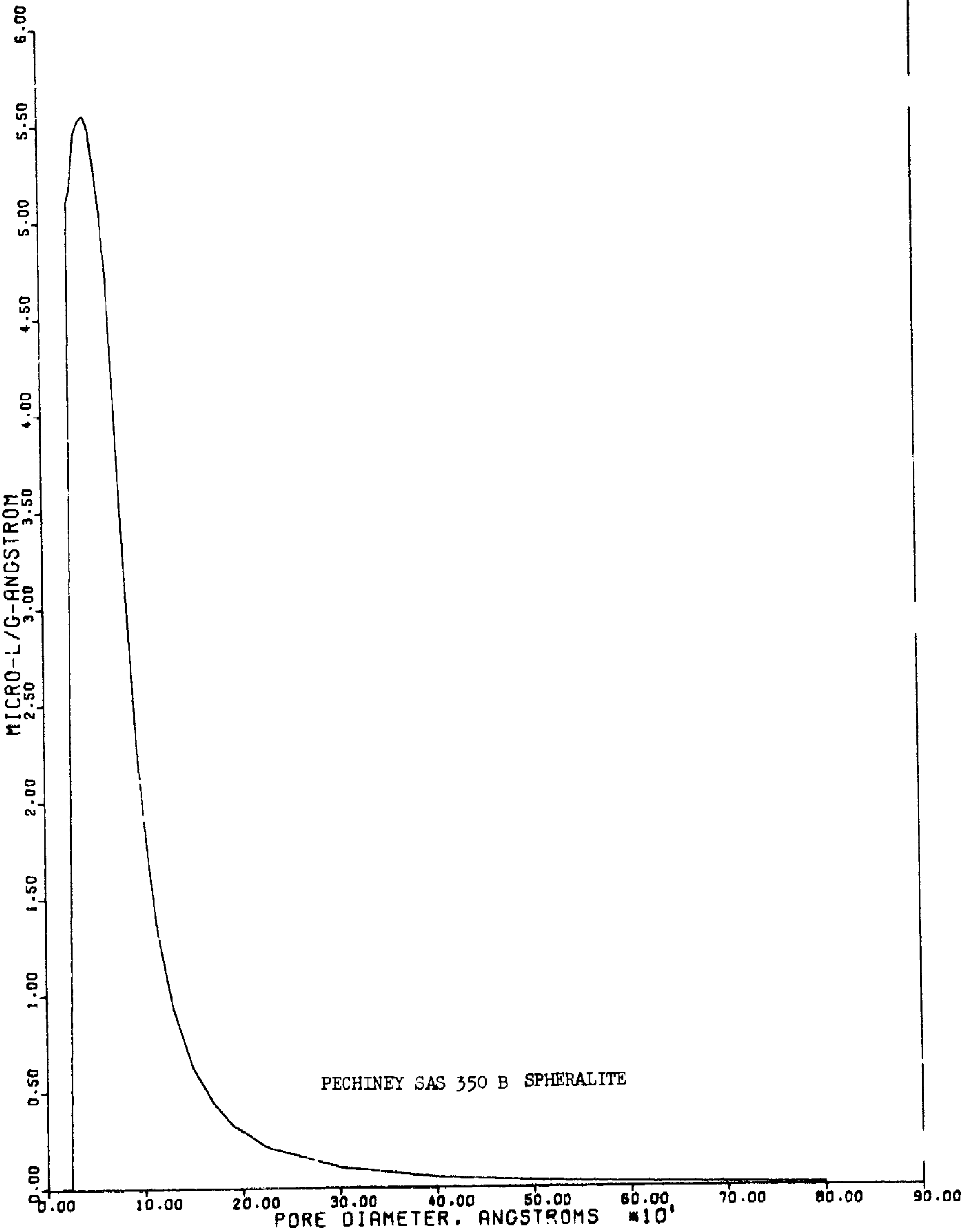



372-A 12724-106
MICRO-L/G-ANGSTROM
6.00
5.50
5.00
4.50
4.00
3.50
3.00
2.50
2.00
1.50
1.00
0.50
0.00
0.00
10.00
20.00
30.00
40.00
50.00
60.00
70.00
80.00
90.00
PORE DIAMETER, ANGSTROMS *10¹
CATALYST 11724-106 ON KA-201 ALUMINA

UNCLASSIFIED

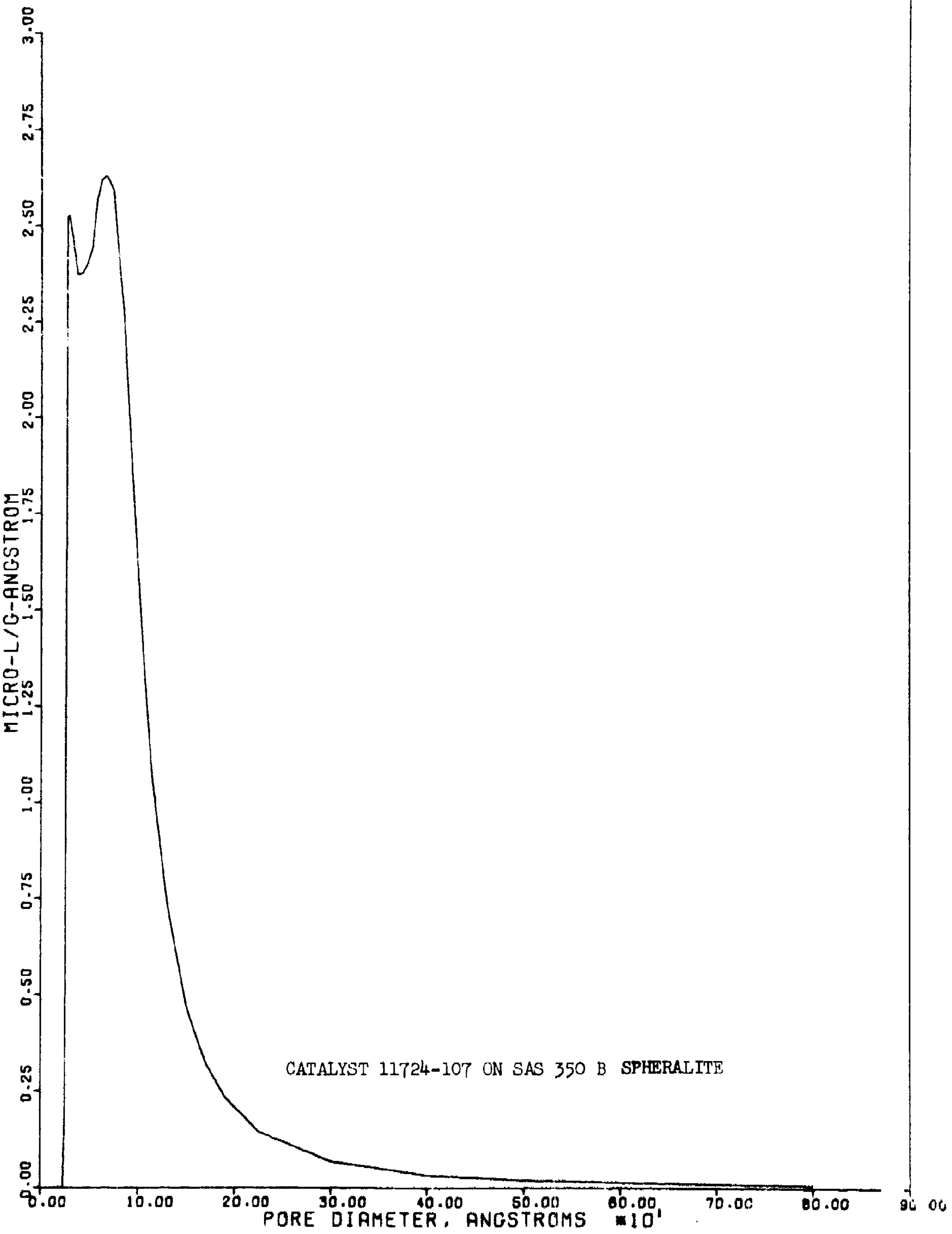
30

372-D 11724-102



UNCLASSIFIED

372-B 11724-107

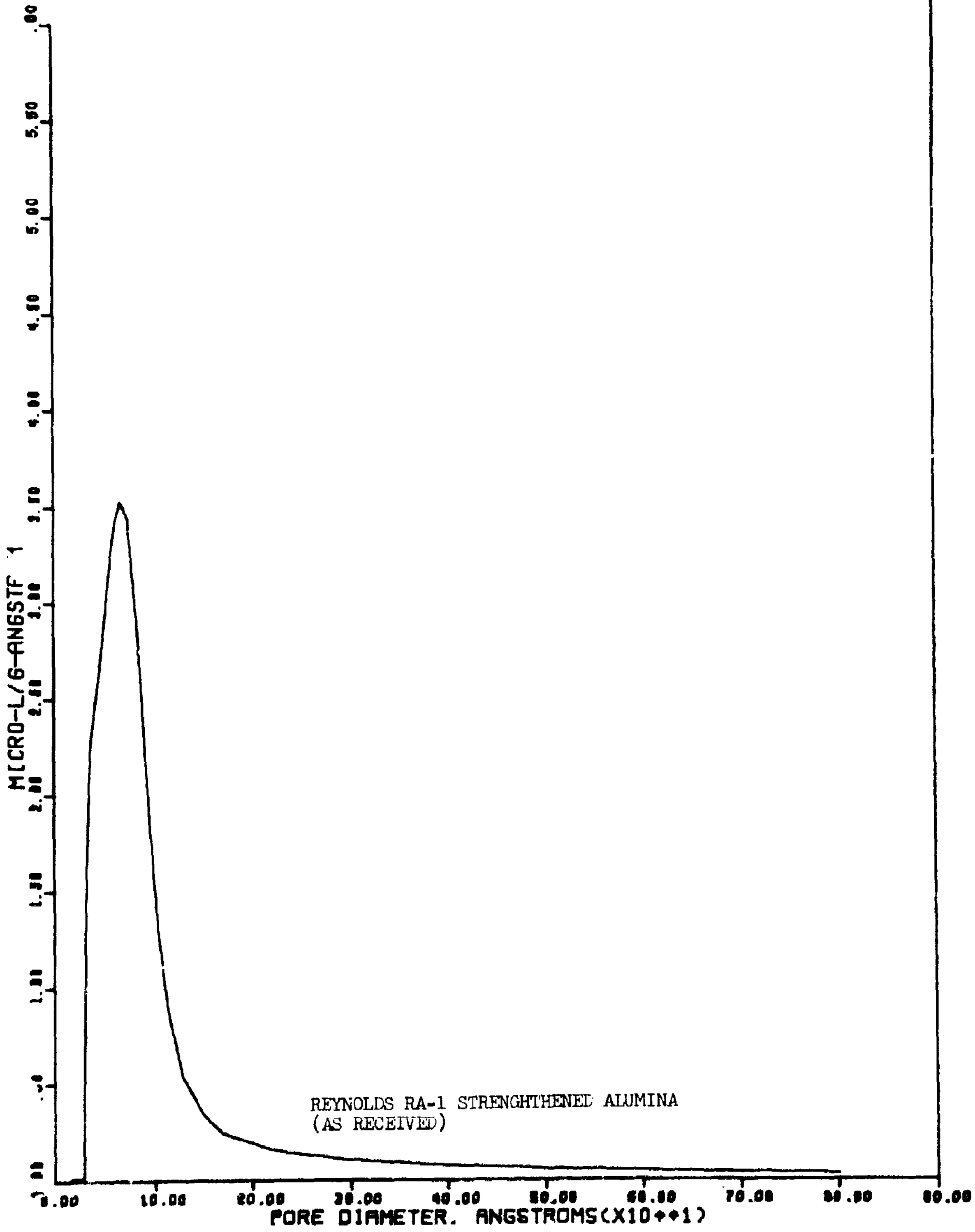


CATALYST 11724-107 ON SAS 350 B SPHERALITE

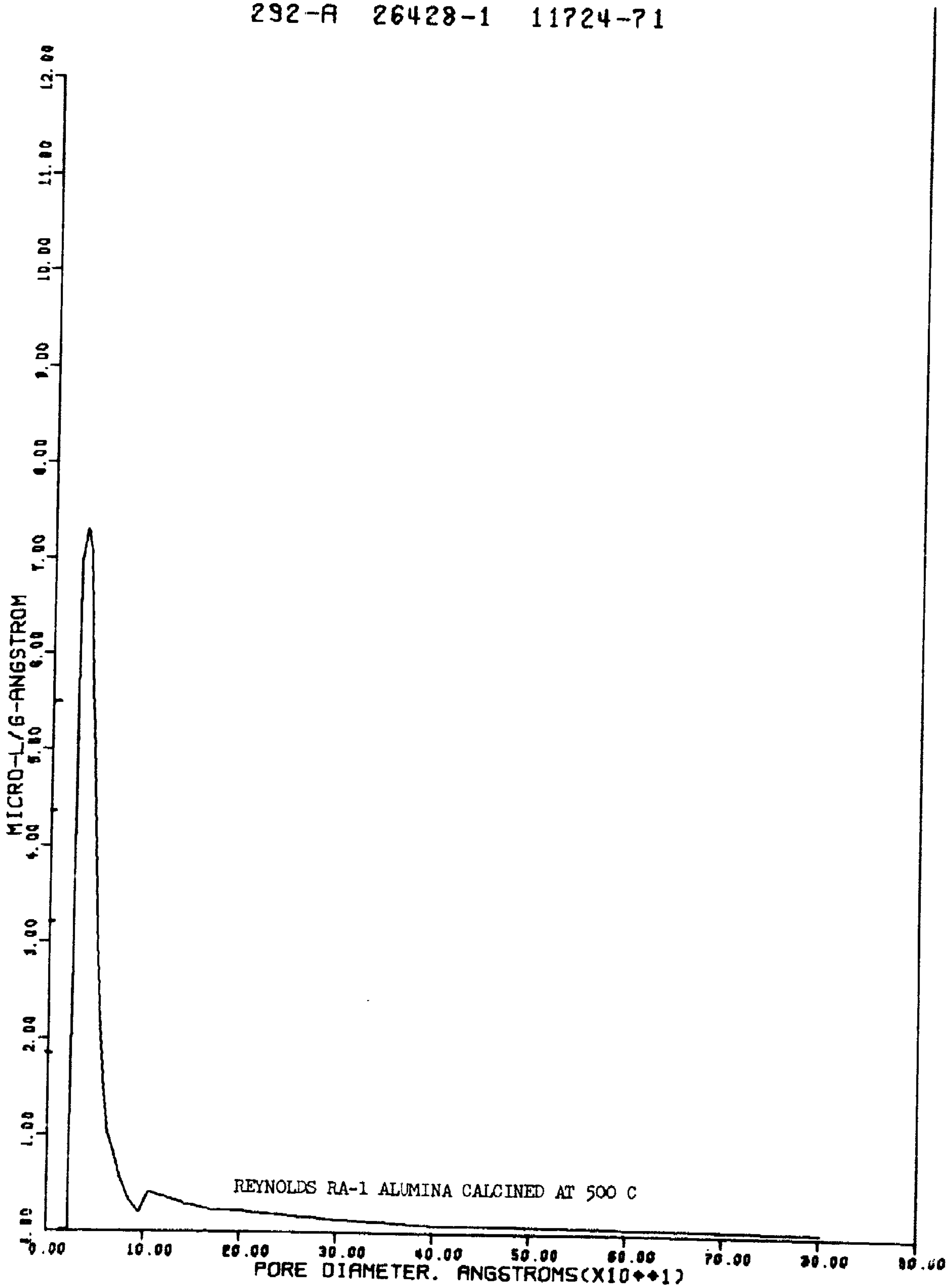
19325

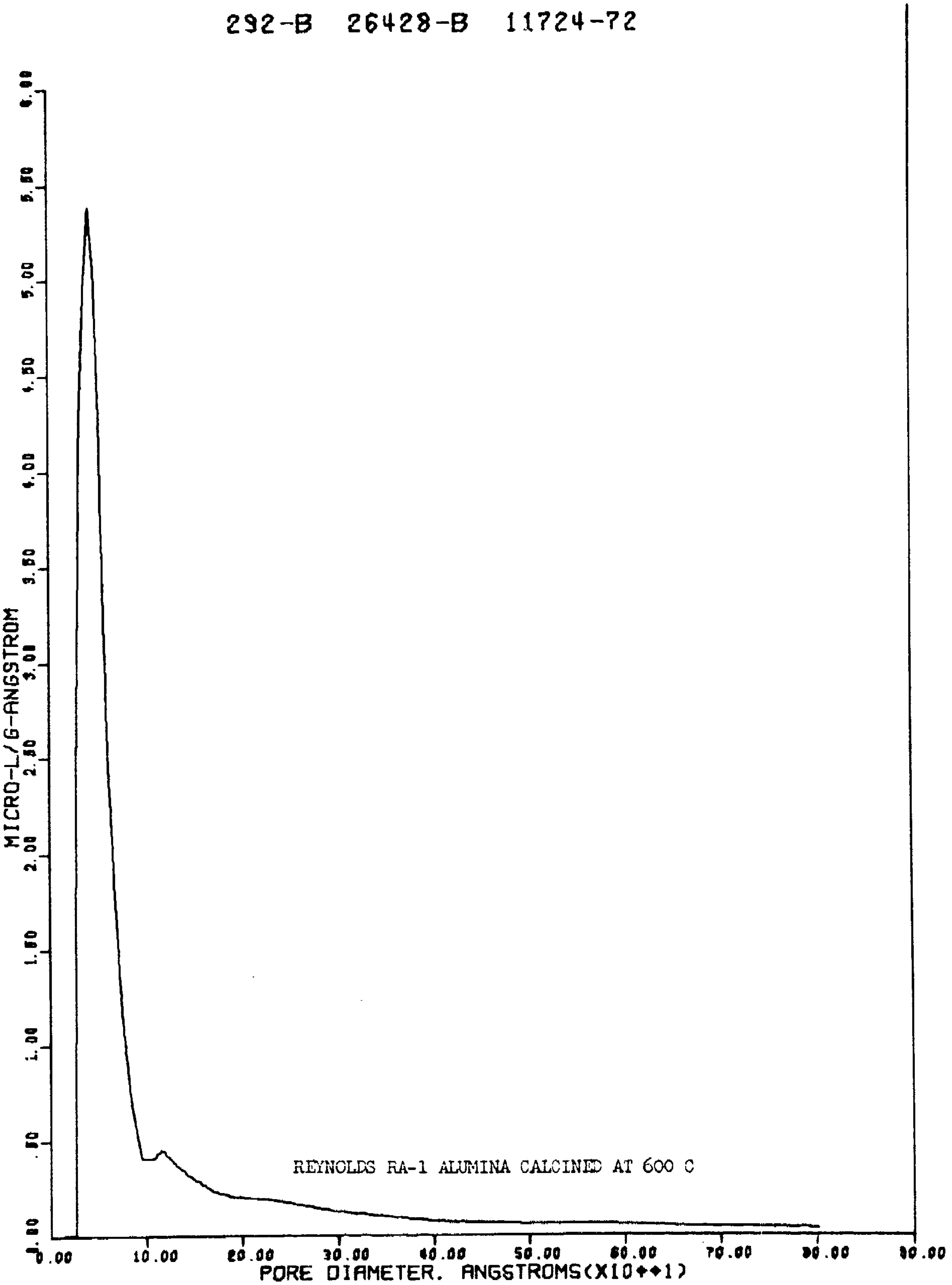
32

220A ~~19925~~ 1 11724 23 ADS



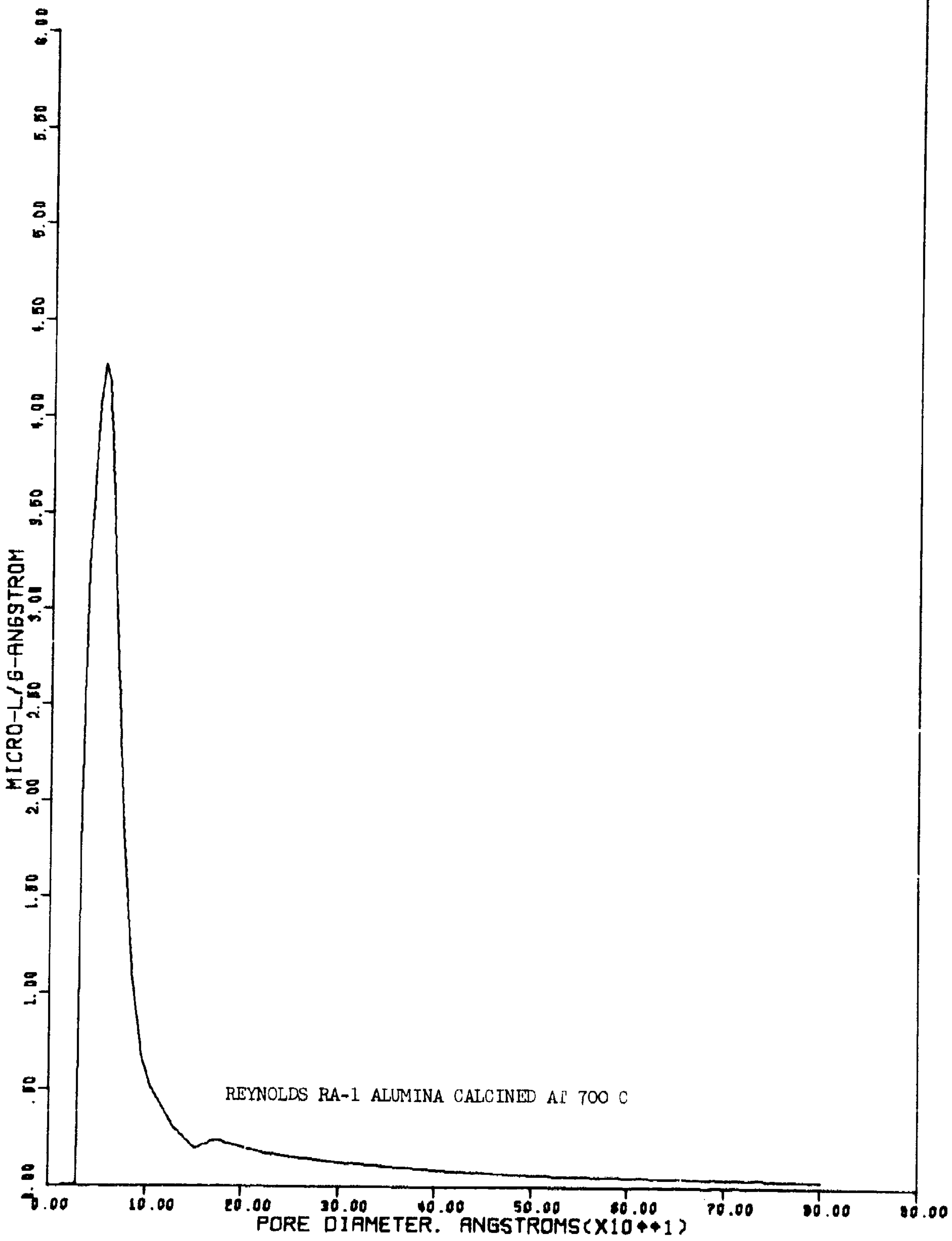
REYNOLDS RA-1 STRENGHTHENED ALUMINA
(AS RECEIVED)


292-A 26428-1 11724-71
MICRO-L/G-ANGSTROM
12.00
11.00
10.00
9.00
8.00
7.00
6.00
5.00
4.00
3.00
2.00
1.00
0.00
0.00
10.00
20.00
30.00
40.00
50.00
60.00
70.00
80.00
90.00
PORE DIAMETER. ANGSTROMS(X10**1)
REYNOLDS RA-1 ALUMINA CALCINED AT 500 C

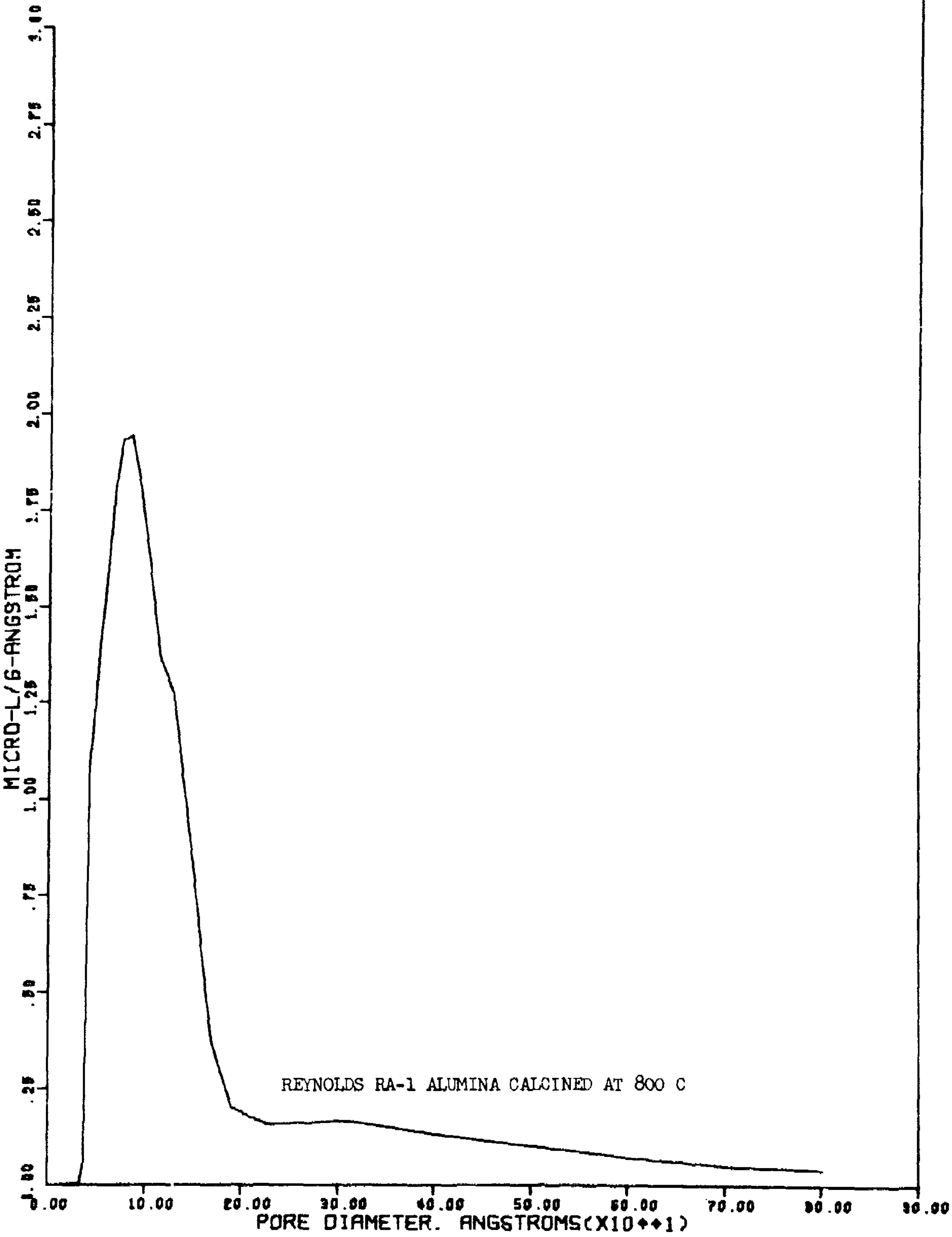

292-B 26428-B 11724-72
MICRO-L/G-ANGSTROM
0.00
0.50
1.00
1.50
2.00
2.50
3.00
3.50
4.00
4.50
5.00
5.50
6.00
0.00
10.00
20.00
30.00
40.00
50.00
60.00
70.00
80.00
90.00
PORE DIAMETER. ANGSTROMS(X10**1)
REYNOLDS RA-1 ALUMINA CALCINED AT 600 C

35

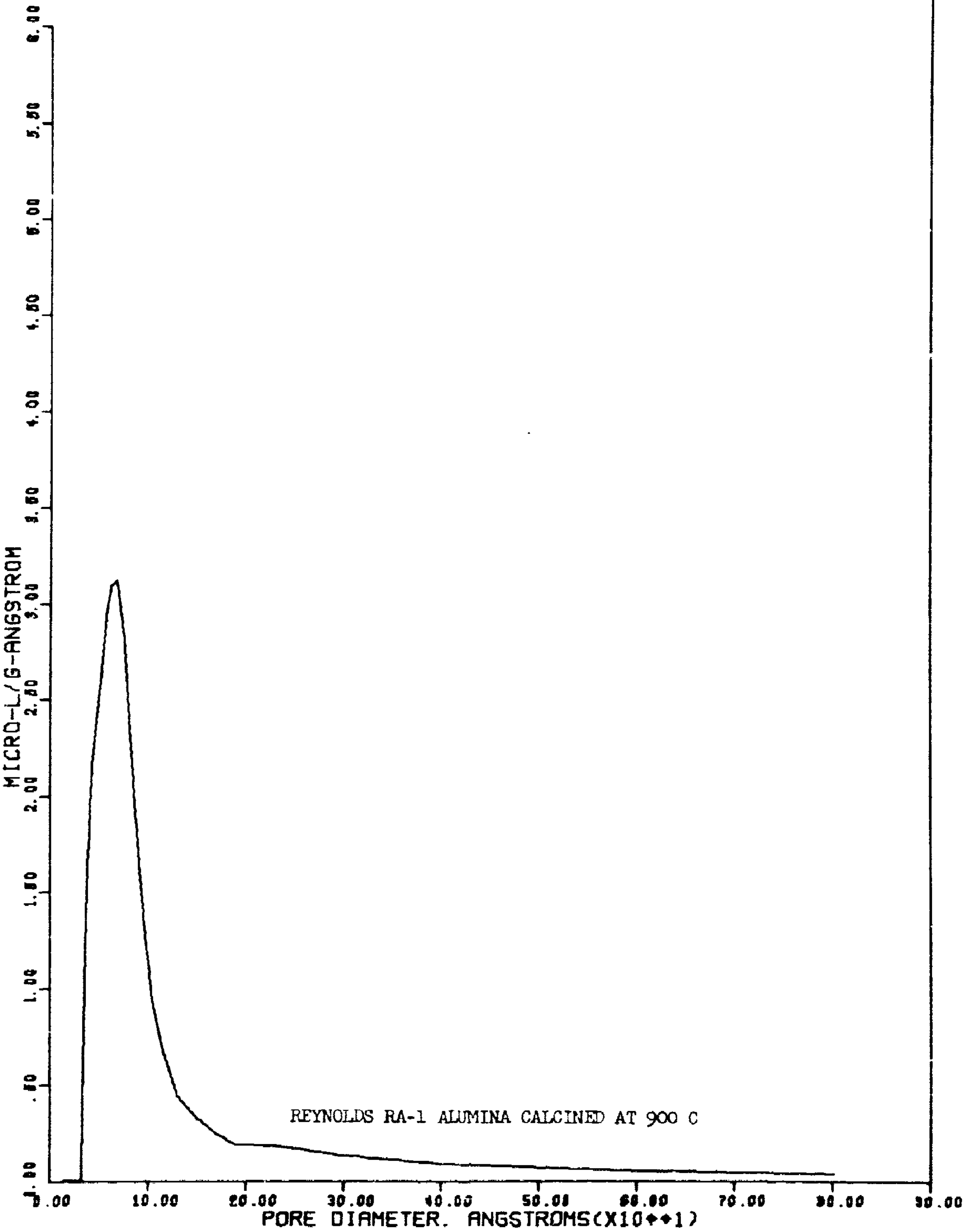
292-D 26428-4 11724-74

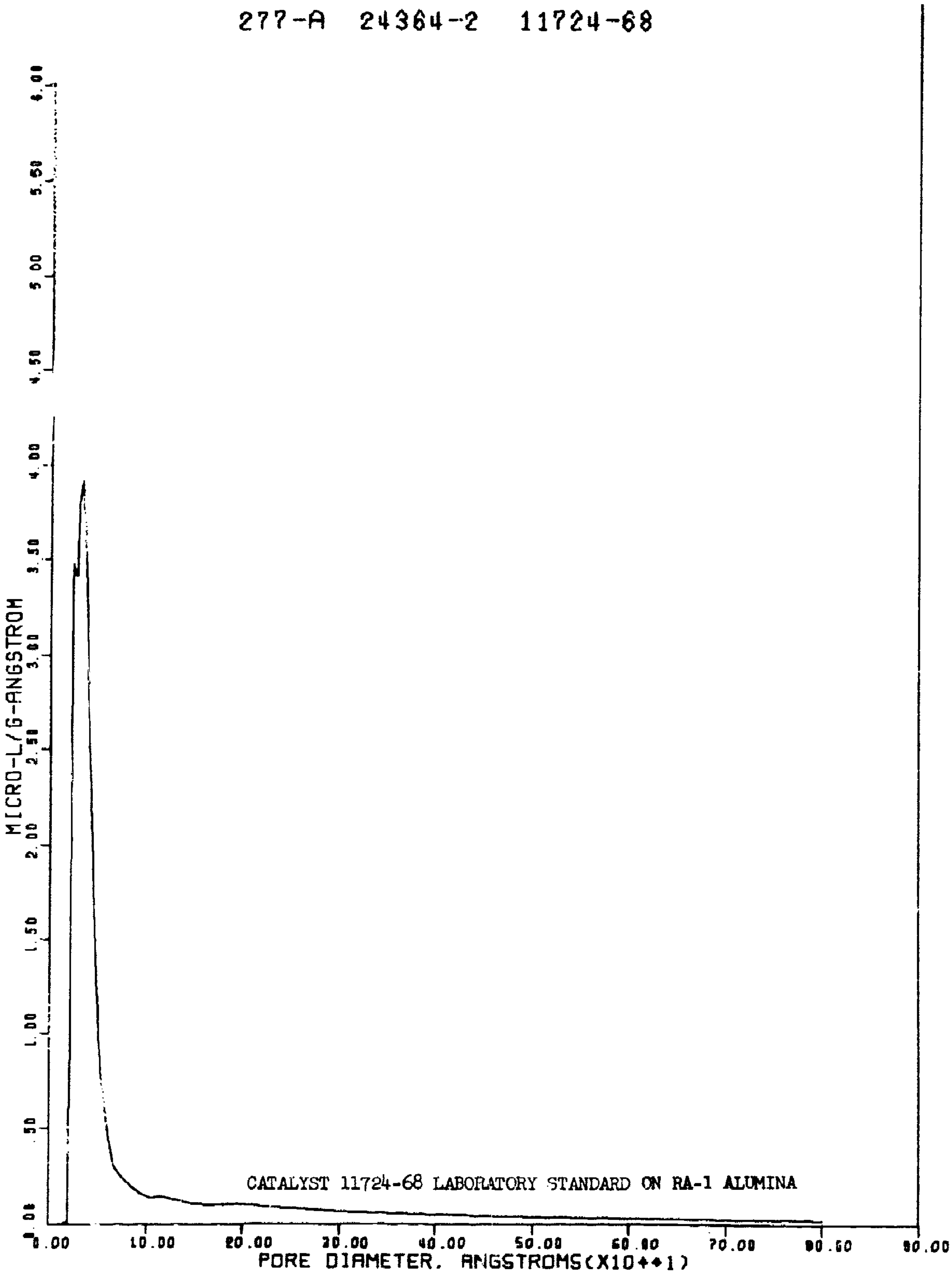


292-C 26428-3 11724-73

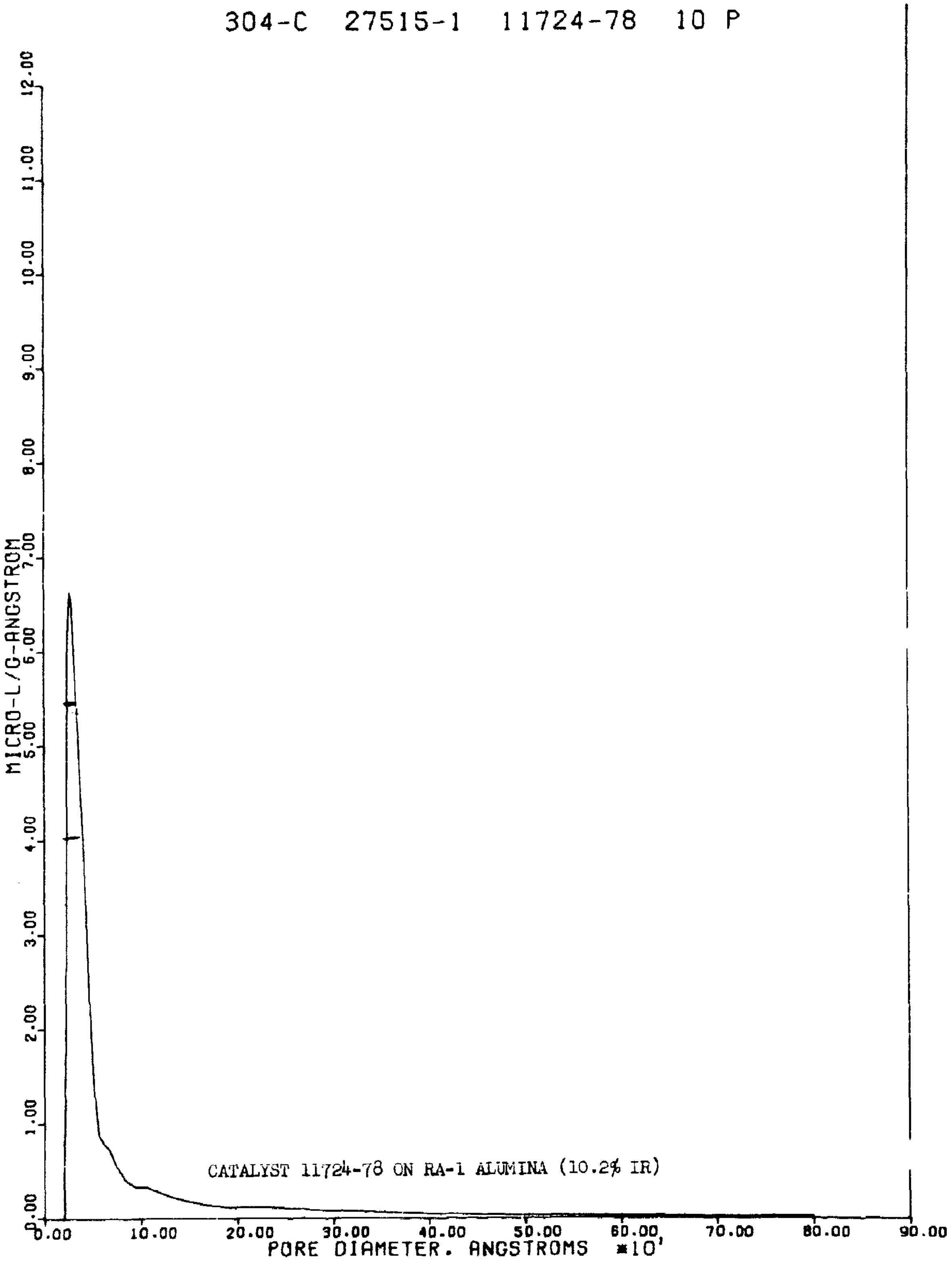


292-E 26428-5 11724-75

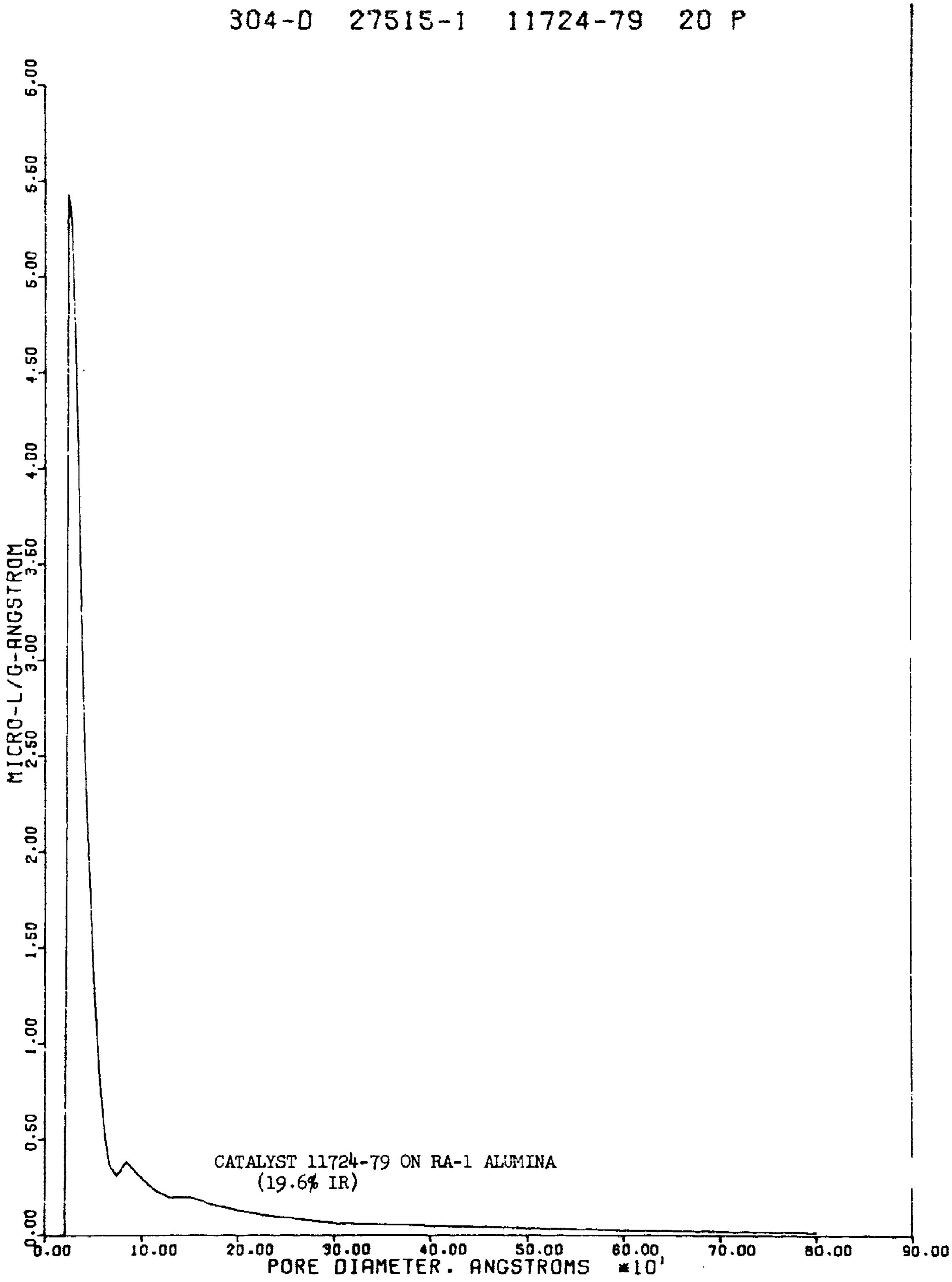




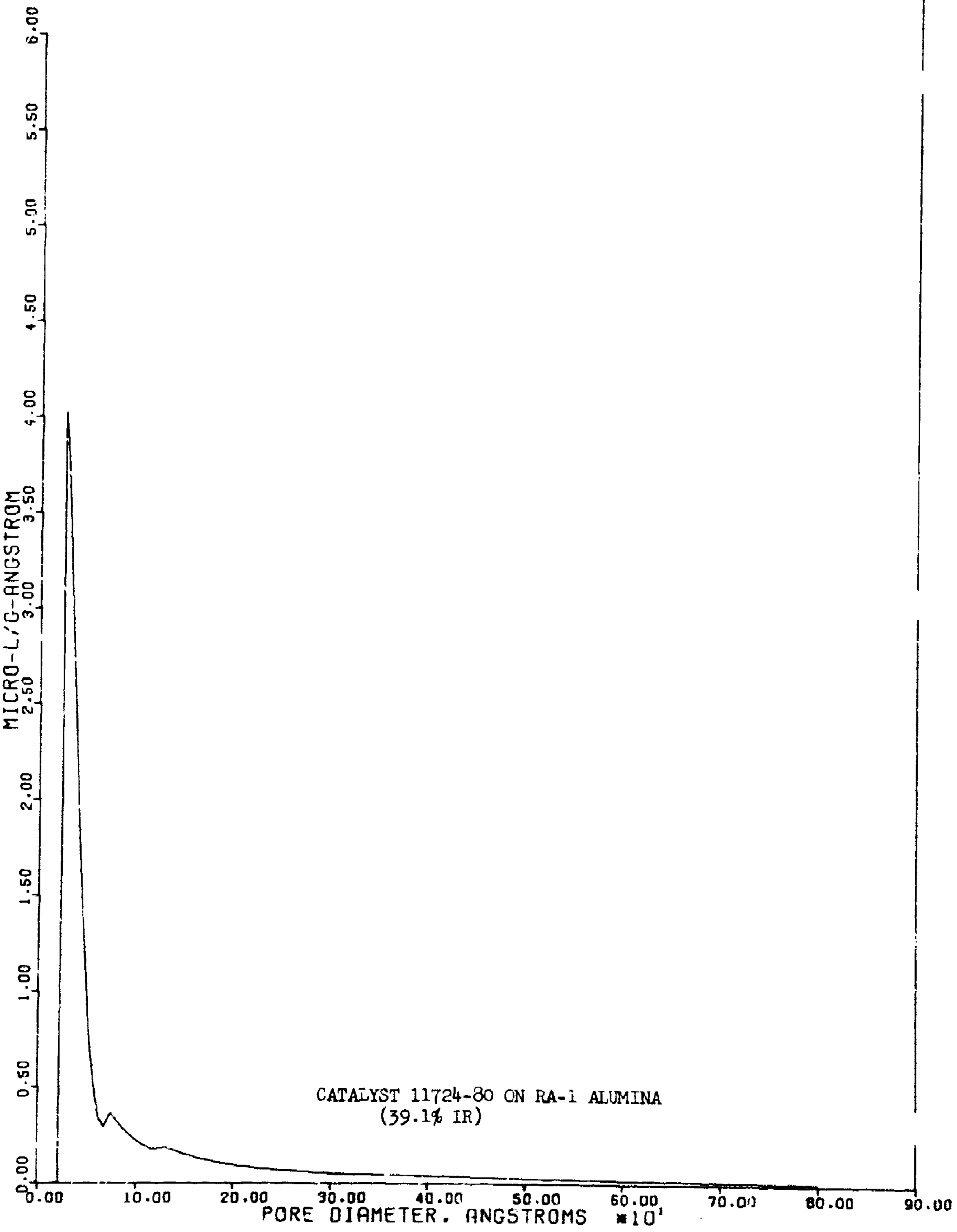
CATALYST 11724-68 LABORATORY STANDARD ON RA-1 ALUMINA



CATALYST 11724-78 ON RA-1 ALUMINA (10.2% IR)

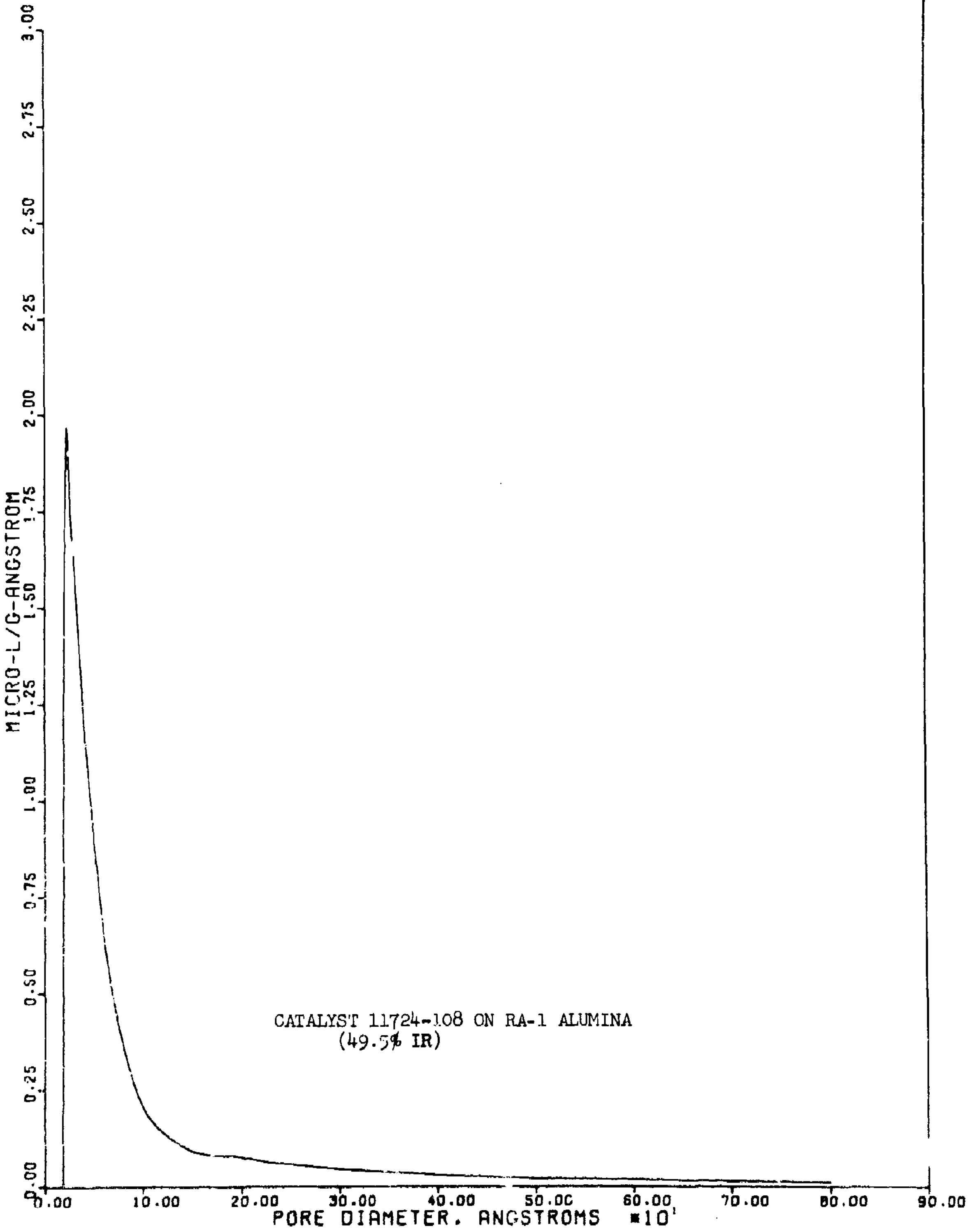

304-D 27515-1 11724-79 20 P
MICRO-L/G-ANGSTROM
0.00
0.50
1.00
1.50
2.00
2.50
3.00
3.50
4.00
4.50
5.00
5.50
6.00
0.00
10.00
20.00
30.00
40.00
50.00
60.00
70.00
80.00
90.00
PORE DIAMETER. ANGSTROMS $*10^{1}$
CATALYST 11724-79 ON RA-1 ALUMINA
(19.6% IR)

304-E 27515-3 11724-80 40 P

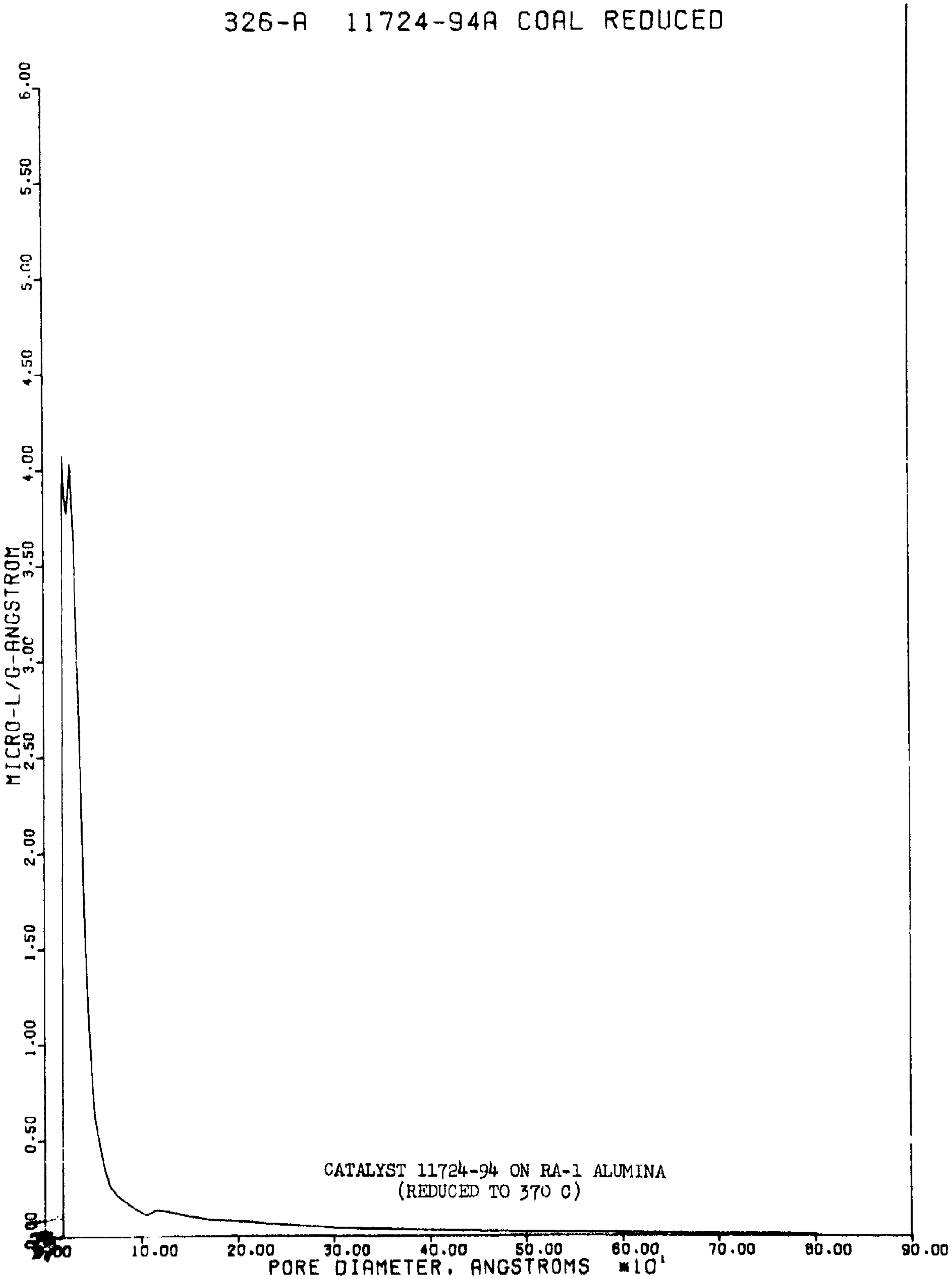


CATALYST 11724-80 ON RA-1 ALUMINA
(39.1% IR)

372-C 11724-108



CATALYST 11724-108 ON RA-1 ALUMINA
(49.5% IR)

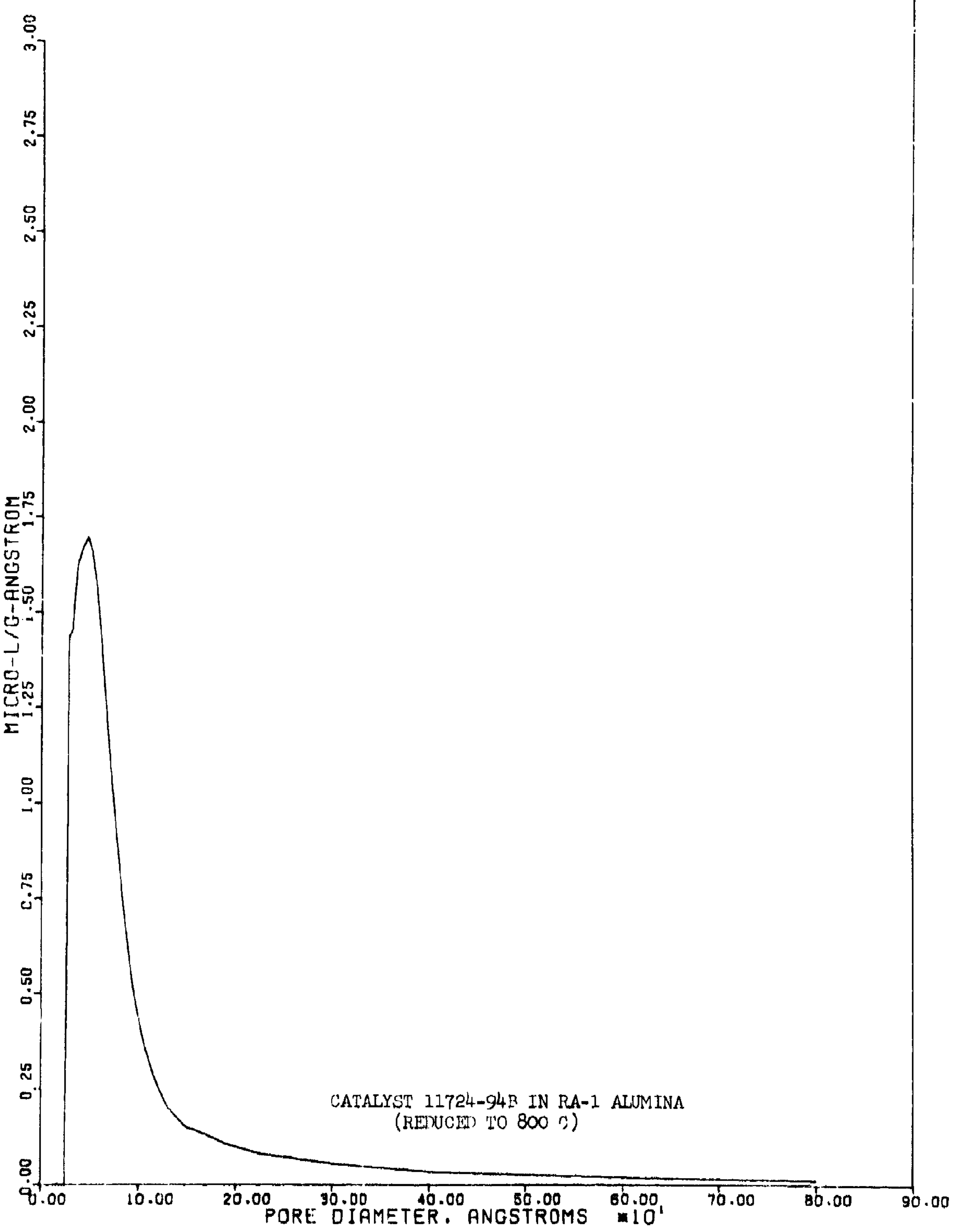


326-A 11724-94A COAL REDUCED

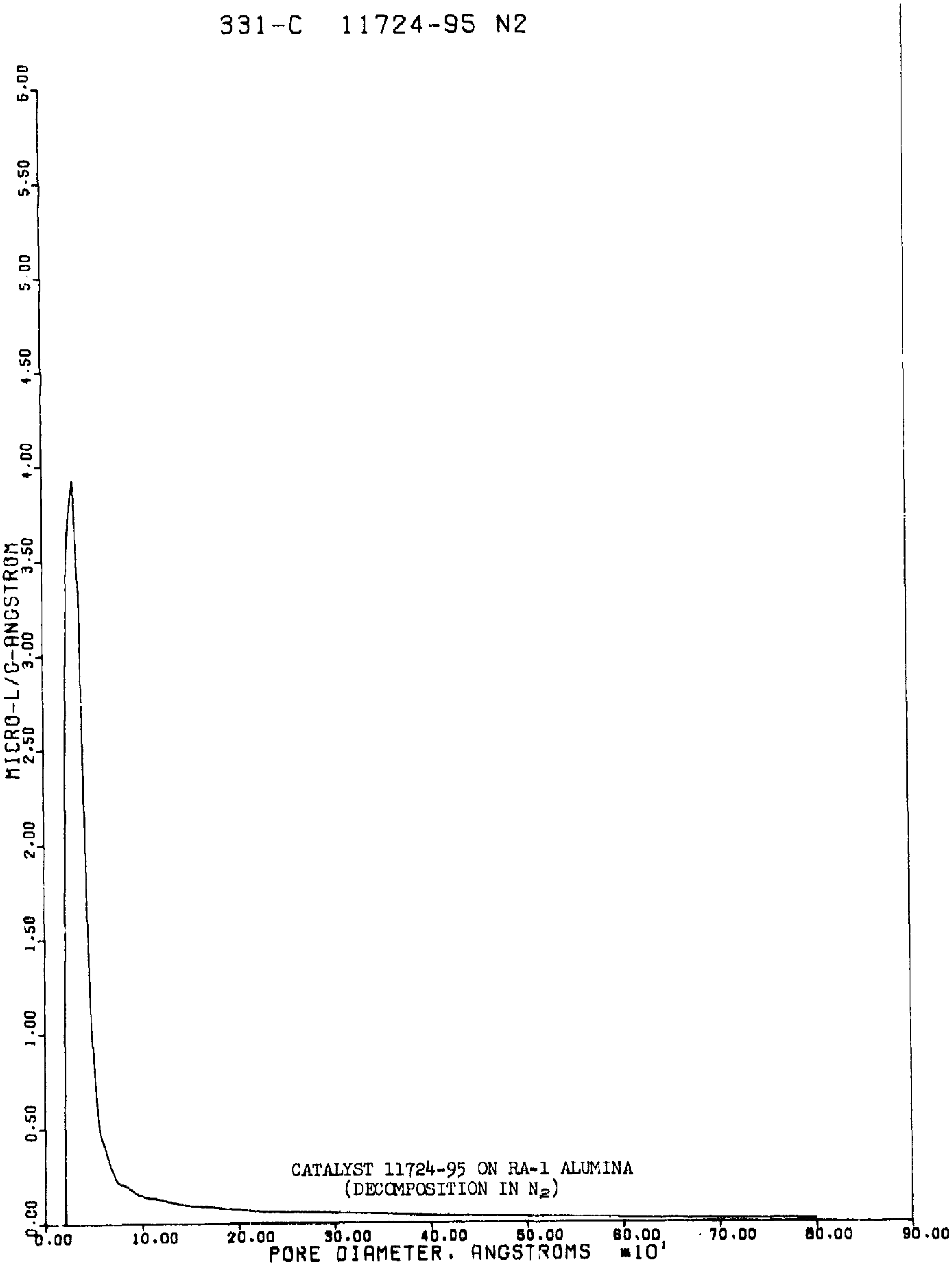
CATALYST 11724-94 ON RA-1 ALUMINA
(REDUCED TO 370 C)

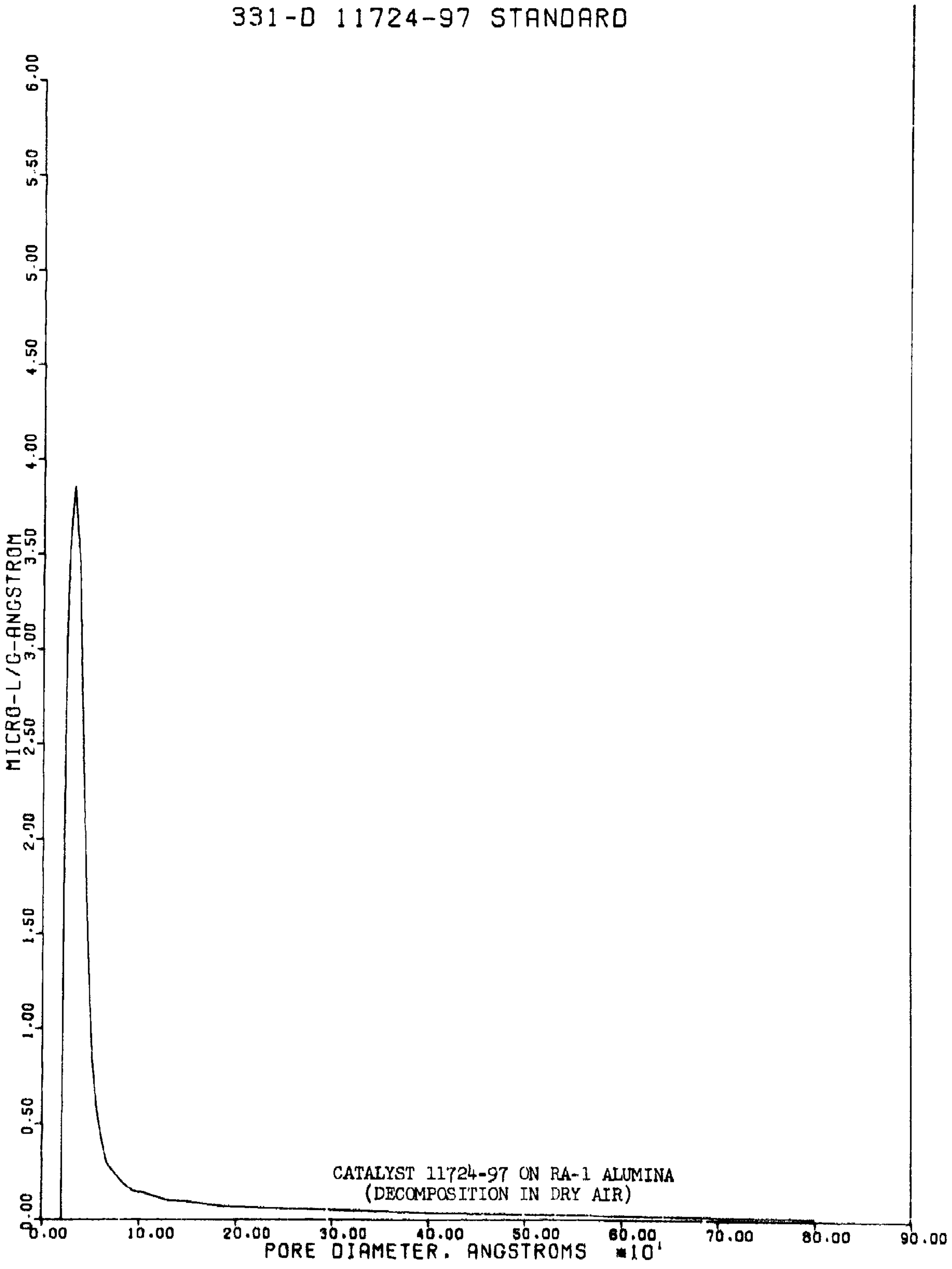
47

326 B 11724-94B 800C REDUCED



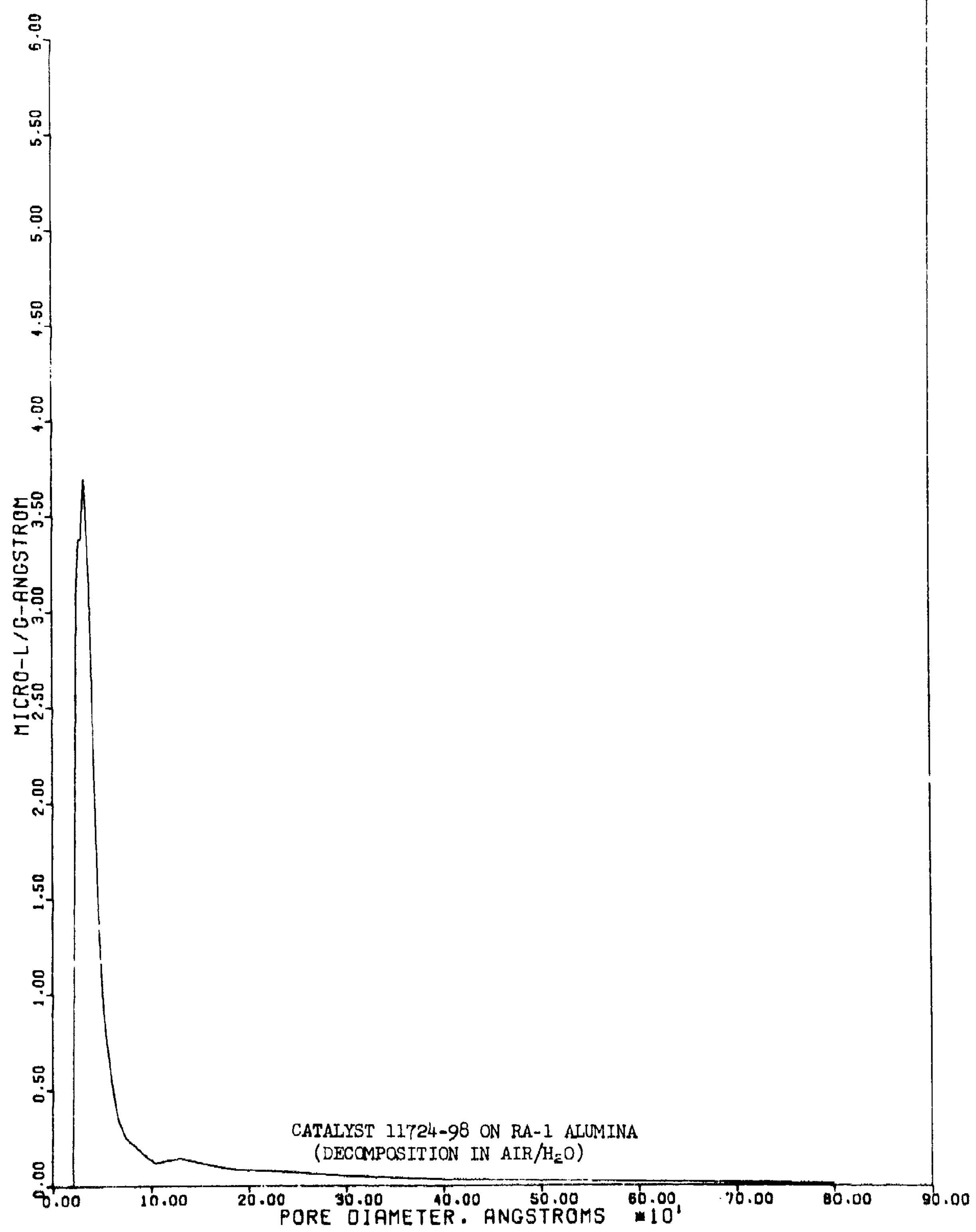
4:


331-C 11724-95 N2
MICRO-L/G-ANGSTROM
0.00
0.50
1.00
1.50
2.00
2.50
3.00
3.50
4.00
4.50
5.00
5.50
6.00
CATALYST 11724-95 ON RA-1 ALUMINA
(DECOMPOSITION IN N_2)
0.00
10.00
20.00
30.00
40.00
50.00
60.00
70.00
80.00
90.00
PORE DIAMETER, ANGSTROMS *10¹

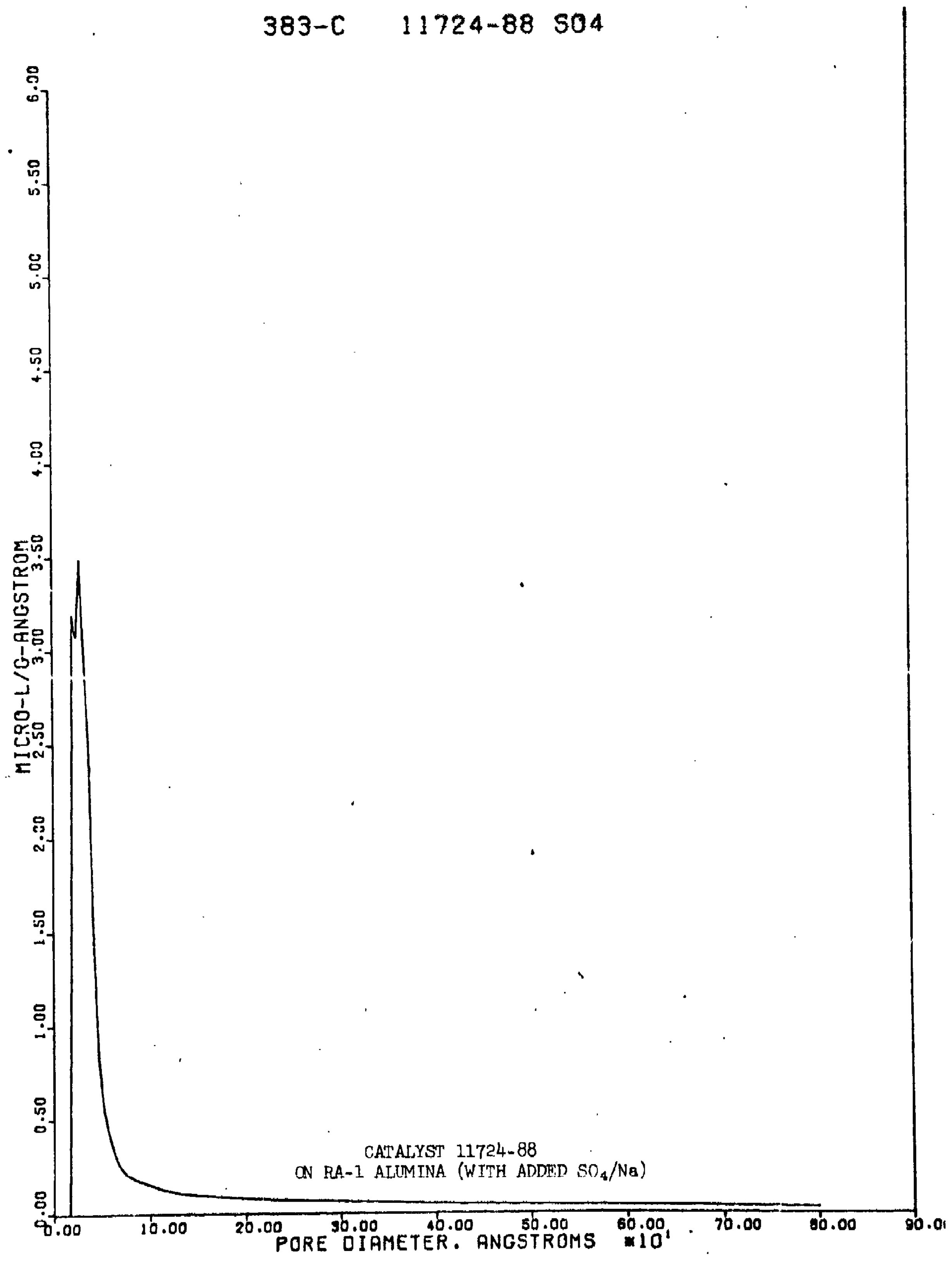

331-D 11724-97 STANDARD
MICRO-L/G-ANGSTROM
0.00
0.50
1.00
1.50
2.00
2.50
3.00
3.50
4.00
4.50
5.00
5.50
6.00
0.00
10.00
20.00
30.00
40.00
50.00
60.00
70.00
80.00
90.00
PORE DIAMETER, ANGSTROMS *10¹
CATALYST 11724-97 ON RA-1 ALUMINA
(DECOMPOSITION IN DRY AIR)

47

331-E 11724-98 WET

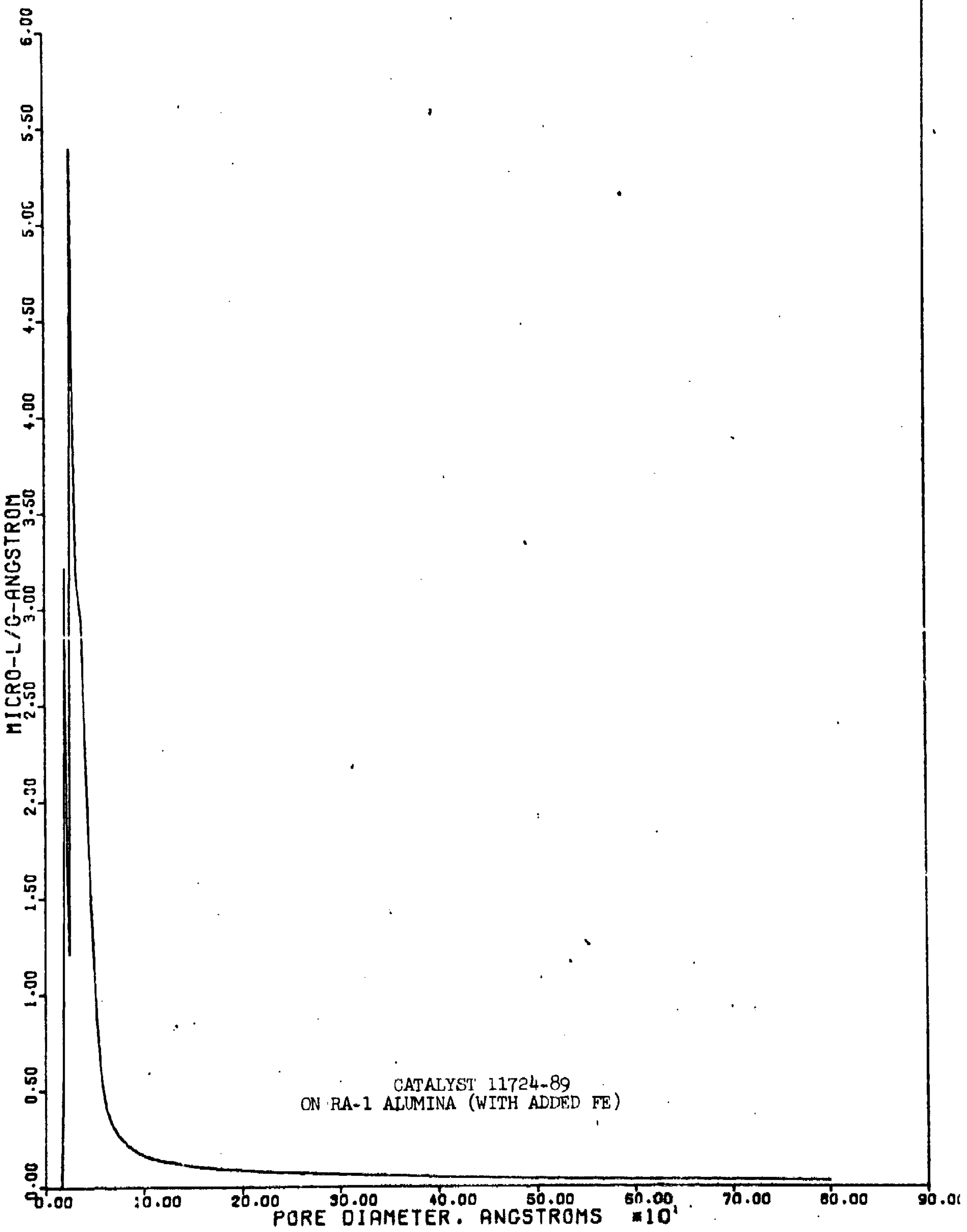


383-C 11724-88 SO4

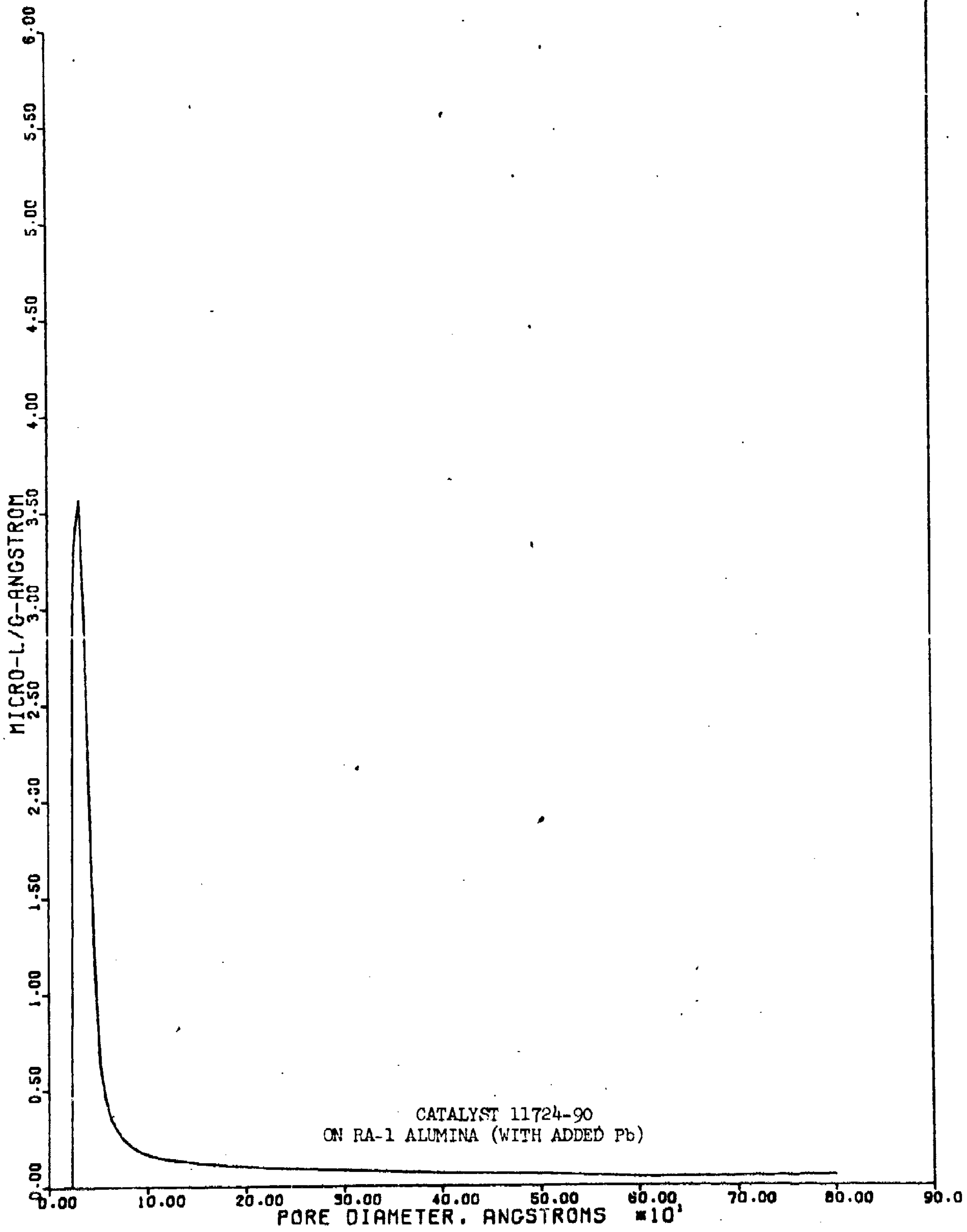


CATALYST 11724-88
ON RA-1 ALUMINA (WITH ADDED SO_4/Na)

383-D 11724-89-FE



383-E 11724-90-PB



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Final Report for period February 1970 - December 1971 2

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10. AVAILABILITY/LIMITATION NOTICES
None

11. SUPPLEMENTARY NOTES	**12. SPONSORING MILITARY ACTIVITY** Air Force Rocket Propulsion Laboratory Directorate of Laboratories Air Force Systems Command United States Air Force Edwards, California

13. ABSTRACT

(U) This report describes a study carried out for the ~~Air Force Rocket Propulsion Laboratory to~~ development of a catalyst of the Shell 405 (Trade Name) type with improved durability to repeated cold starts using monopropellant hydrazine. Catalysts that differed from standard Shell 405 (Trade Name) Catalyst in respect to composition and properties of the support material and to the conditions of catalyst preparation, iridium content, and concentrations of certain normal trace impurities were prepared for testing ~~at the AFRPL~~.

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14. KEY WORDS

Shell 405 Monopropellant Hydrazine Catalysts
Product Improvement
Substrates
Physical Property Variation
Processing Techniques