SLAC-TN-64-5
Earl Hoyt
January 1964



# EFFECT OF SURFACE TREATMENT AND BAKING ON THE OUTGASSING CHARACTERISTICS OF 304 STAINLESS STEEL PIPE

## ABSTRACT

Relative outgassing rates and pumpdown performance data were obtained for type 304 stainless steel pipe after various surface treatments, vacuum bakeouts, and air and nitrogen exposures. The results of these tests indicate that outgassing rates below one pico-torr liter/sec/cm$^2$ can be obtained with baked piping. Furthermore, these low outgassing rates can be re-established without further baking after exposing baked piping to air or nitrogen.

# I. INTRODUCTION

The final design of the two-mile accelerator calls for stainless steel vacuum manifolding having an internal surface area in excess of sixty-four million square centimeters. The copper accelerator tube and related waveguides will have an internal area of near twenty-two million square centimeters.

In this essentially all-metal "leak-free" system, the pumping requirements are predicated on the basis of expected outgassing rates of the stainless steel and copper internal surfaces.

As part of a more general program to establish outgassing rates for these materials, we undertook to determine what effect different surface treatments had on the outgassing behavior of commercial stainless steel pipe, type 304.

The following questions were asked:

1. What surface treatment will yield the lowest relative outgassing rates?

2. What (if any) bakeout schedules are necessary to achieve low room temperature outgassing rates?

3. What (if any) change in outgassing rate results after exposing baked piping to air or nitrogen?

4. How is pumpdown performance influenced by air or nitrogen exposure?

5. Does time of exposure effect subsequent pumpdown behavior?

## II. LITERATURE SURVEY

A brief literature survey revealed the following pertinent experimental outgassing data:

Griessel[1] measured the quantity of gas evolved from type 305 stainless steel when heated in vacuum to 1000°C, utilizing three different treatments.

A. Trichlorethylene degreased, alkali boiled.

B. Vacuum fired (30 minutes at 1000°C at $10^{-5}$ torr).

C. Hydrogen fired (30 minutes at 1000°C) plus vacuum fired as B.

The results showed little, if any, advantage to the intermediate hydrogen treatment. The surface-cleaned sample released about six times as much gas as the two vacuum fired samples (B and C).

Morrison[2] obtained similar results when measuring the quantity of gas removed from high purity nickel following surface cleaning, heating in vacuum, heating in hydrogen and heating in forming gas. All heat treatments including vacuum (pressure not given) increased the total gas content.

Varardi[3] measured the effect of surface pretreatment on the degassing behavior of nickel. Varardi found little difference in the total amount of gas released from previously hydrogen-fired samples compared with acid-cleaned samples. His results indicate that samples only degreased released more than ten times as much gas as the hydrogen or acid treated samples.

Further measurements of the degassing properties of various grades of nickel have been reported by Lawson.[4] Lawson found that the total quantity of gas evolved, when heating to 975°C, is limited by the amount of oxygen present in the form of oxides. In impure ($\approx$ 99.8%) nickel these oxides were found to be concentrated near the surface as grain boundary impurities. In this case the evolution of CO (the principal gas evolved) is controlled by the rate of carbon diffusion. In the case of purer nickels (> 99.99%) the gas evolution is slower and is controlled by the diffusion rate of the oxygen in the nickel lattice. The total gas content of the more impure nickels was significantly reduced by etching-off a 15μ thickness from the surface of the samples.

Outgassing rates for stainless steel have been reported by Holland,[5] Power and Robson,[6] and Das.[7]

Holland reports an outgassing rate of 2 pico-torr liters/sec/cm$^2$ for a stainless system, 95 percent of which had been baked for over 3-1/2 hours at 350 to 400°C.

Power and Robson report values for electropolished stainless steel before and after baking as follows:

| History | Outgassing Rate<br>Pico-torr liters/sec/cm$^2$ |
|---|---|
| After 24 hours pumping at room temperature | 200 |
| Baked for 16 hours at 400°C - then temperature was reduced in stages to ambient | 0.3 |

They found the baked samples remained highly temperature sensitive at all temperatures below the bake temperature. Using their data, one can compute a temperature coefficient of outgassing at 0.02 pico-torr liters/sec/cm$^2$/°C.

Das measured the outgassing characteristics of a type 304 stainless steel bell jar with the surface in the rough ground condition. He reports a room temperature outgassing rate of 0.93 pico-torr liters/sec/cm$^2$ after baking for over 80 hours at 400°C. Before bakeout he reports a rate of 110 pico-torr liters/sec/cm$^2$ after pumping for 167 hours.

Flecken and Nöller[8] measured the total amount of gas evolved at 450°C from three different stainless alloys following three different surface treatments:

A. No treatment specified

B. Steam degreased

C. Vacuum fired for 1 hour at $10^{-3}$ torr

In general the vacuum fired samples released about one order of magnitude less gas than did the steam degreased samples, which in turn released only half as much gas as did the untreated samples.

With regard to pumpdown performance following various exposures, the work of Griessel,[1] Varardi,[3] Das,[7] Hayashi,[9] and Bowden and Throssell[10] deserve mention.

Griessel maintains that "contaminants from the surrounding atmosphere are readily absorbed on clean parts, especially porous ones. Evidence of contamination by cleaning solvents has been found on parts which have passed through the room where these solvents are in use, although the parts have never come in direct contact with them." He also states that "adsorption of water from the atmosphere is very rapid and seriously limits pumpdown time, especially of porous materials. In some cases it is tenaciously held on a surface and may persist at very high temperatures."

Varardi showed that storage conditions (wet or dry) have little influence on subsequent outgassing behavior. However, he found handling techniques to be critical. Samples touched with fingers, cotton gloves, or rubber cots increased greatly in total gas content.

The results reported by Das are somewhat anomalous in that he found lower outgassing rates for stainless steel after venting to moist air than he did after venting to dry nitrogen. His results also show some increase in outgassing rates following air and nitrogen exposure.

Hayashi reported the time to evacuate (to $10^{-5}$ torr) a vessel vented to moist air was a factor of two hundred over that required to reach the same pressure when the vessel was vented to dehumidified air.

Bowden and Throssel made very careful measurements of water and organics adsorbed on gold and platinum surfaces. Their experiments show that "clean" metal surfaces adsorb no more than one or two monolayers, even at 90 percent of saturation pressure. However, they found that rinsing the samples in tap water before evacuation and remeasurement contaminated the surfaces to the extent that they adsorbed the equivalent of 48 molecular layers of water. They maintain that the heavy adsorption reported in the literature is attributable to uncertainties about surface areas or, more likely, to the presence of surface contaminants.

## III. EXPERIMENTAL METHOD AND ERRORS

Four methods of pretreating the stainless steel surfaces were studied to determine relative outgassing rates and relative pumpdown performance.

The four methods were:

1. Electropolishing
2. Hydrogen firing
3. Chemical cleaning
4. Glass bead blasting

Some variations of these treatments were also tried and will be discussed later in the report. Details on the surface treatments are listed in Table I in the appendix. The outgassing test system is shown schematically in Fig. 1. With the exception of the glass-enveloped ion gauge, the system was all metal.

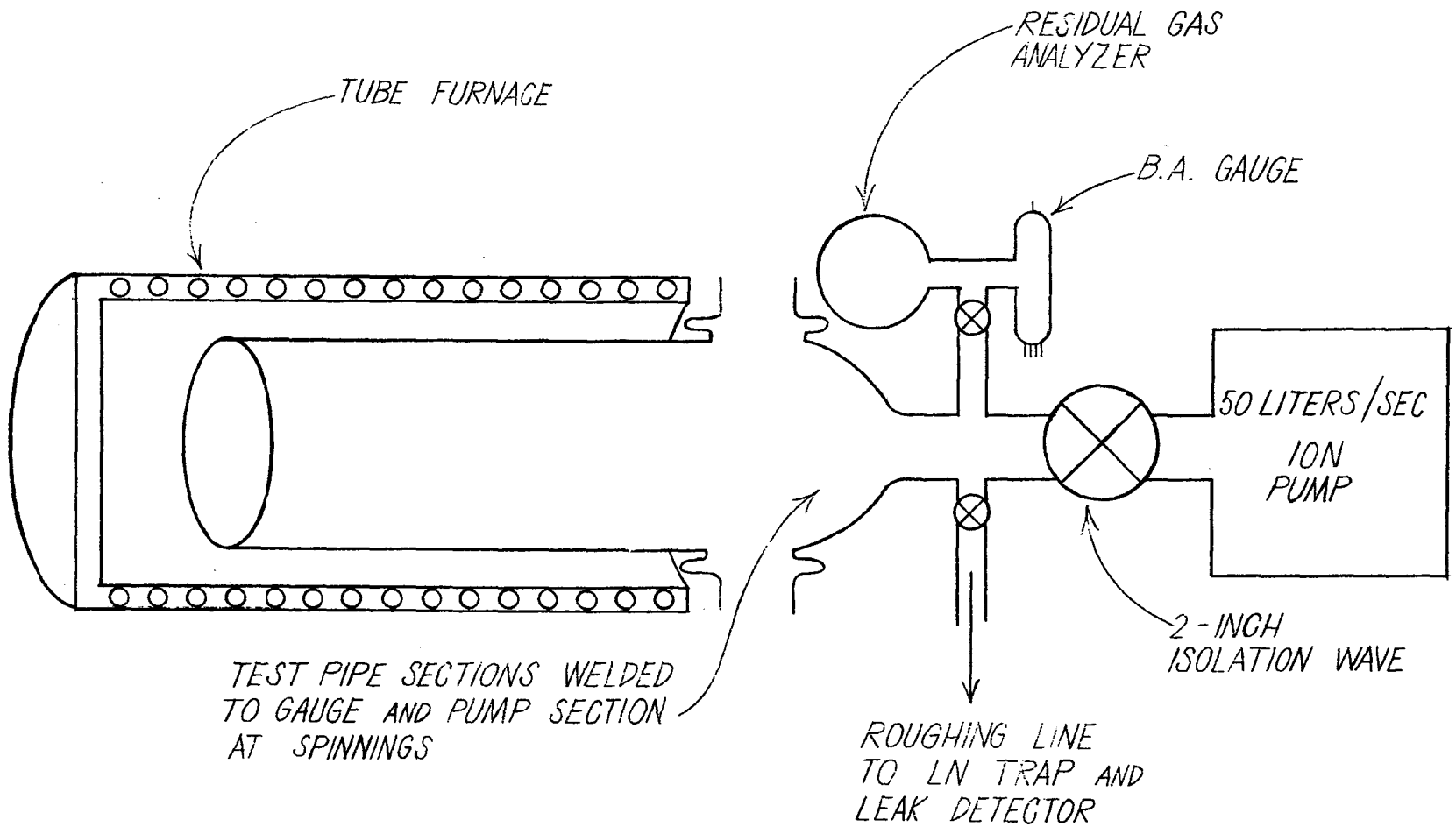
The test pieces were fabricated from twenty-inch sections of 6-inch, (6.4-inch I.D.) schedule 5, type 304 stainless steel pipe. One end of the pipe was welded closed by means of a plate, and the other end was welded to a spinning to ensure easy, clean welding to the gauge and pump section of the test rig. By means of the spinnings it was possible to weld on a test pipe, make a set of measurements, grind-off the weld, and weld on another test pipe. This process could be repeated many times. Each time the diameter of the spinning at the weld was reduced by about 1/8-inch.

Commercial stainless steel pipe from two sources was tested, although most of the tests were conducted on sections cut from a single length of 6-inch pipe. The chemical analysis of the two alloys used is listed in Table II in the appendix.

The first measurements were made on the gauge section of the test rig. This was done by blanking-off the spinning with an SS plate. The gauge section was then baked under vacuum at $400^{\circ}C$ for two days and the outgassing rate was measured. Outgassing rates were measured by the pressure rise method, wherein the system is isolated from the pump and the pressure increase is recorded as a function of time. The slope of this curve is then taken as the rate of pressure rise. Knowing the volume and the surface area, one computes the desorption rate as:

$$D = \frac{dP}{dt}\ \frac{V}{A}\ \text{torr-liters/sec/cm}^2$$

where $P$ is in torr, $V$ is in liters, $A$ is in $cm^2$, $t$ is in seconds.


RESIDUAL GAS ANALYZER
TUBE FURNACE
B.A. GAUGE
50 LITERS/SEC ION PUMP
2 - INCH ISOLATION WAVE
TEST PIPE SECTIONS WELDED TO GAUGE AND PUMP SECTION AT SPINNINGS
ROUGHING LINE TO LN TRAP AND LEAK DETECTOR

Having determined the outgassing rate contribution of the gauge section, we felt we could proceed to measurements on the test pieces. This was a naive assumption, as we learned shortly after baking the first test pipe. The total desorption of the system was found to be lower than that measured earlier for the gauge section alone. This meant that only relative outgassing rates could be measured. The volume of gauge section was $\approx$ 4 liters, whereas the volume of the total system was $\approx$ 14 liters. The area of the gauge section was $\approx$ 1400 $cm^2$ and the total system area, exclusive of pump, was $\approx$ 4500 $cm^2$.

In view of the above, it was decided to compute total outgassing rates using the total area and to assume that the outgassing rate was the same for the gauge and test sections. In all probability the outgassing rate values reported here are somewhat higher than the real ones; however, the prime purpose of this study was to make relative measurements and this was done with minimum error.

Outgassing rate measurements were made intermittently during all phases of a run. This was accomplished by closing the isolation valve to the pump and recording the pressure increase, measured with the Bayard-Alpert ion gauge, on one channel of a two-channel recorder. The other channel of the recorder was used to record partial pressures from the output of the residual gas mass spectrometer.* In this way a continuous record of total and partial pressures was made during one run with each of the main surface treatments. Unfortunately, a mass spectrometer was not available continuously, and some of the runs were made without benefit of partial pressure analysis.

---

* Associated Electrical Industries MS-10 mass spectrometer. Loaned through the courtesy of Mr. Al Bocek of Sicular Industrial X-Ray Co.

## IV. RESULTS AND DISCUSSION

The first four runs were made using an initial 60-hour schedule, as follows:

| | |
|---|---|
| Room temperature pumpdown | 24 hours |
| Heating to 400°C | 8 hours |
| Hold at 400°C | 16 hours |
| Cool to 200°C | 2 hours |
| Hold at 200°C | 8 hours |
| Cool to room temperature | 2 hours |

The pipes were then exposed to air or nitrogen and again pumped down. The results of the first four 60-hour runs are shown graphically in Figs. 2 and 3.

Residual gas analysis showed that both the hydrogen-fired pipe and the electropolished pipe were releasing more hydrogen and hydrocarbons than were the chemically cleaned and bead blasted pipes. Qualitative results of the residual gas analysis are summarized in Table III. After comparing the results obtained on these pipes, it was decided to concentrate further work on chemical cleaning and glass bead surface treatments.

Figures 4, 5 and 6 depict the outgassing rates measured for three pipes having slightly different chemical surface treatments.

Pipe #2 was given an extended room temperature pumpdown in an attempt to measure how long it took for the outgassing rate to flatten out. It can be seen that the rate changed very little after 48 hours.

In comparing Figs. 5 and 6, where the same pumpdown-bakeout schedule was followed, it is apparent that the etched pipe released less total gas and gave the lowest outgassing rate after bakeout. It would appear that some surface removal does indeed reduce the total gas content disproportionately, as was suggested by the work of Lawson.

Figures 7 and 8 show the outgassing rates determined for glass bead honed pipes #2 and #3. The difference here was that pipe #2 was chemically cleaned prior to the honing, whereas pipe #3 was only degreased prior to honing. If one were to make a judgment on the basis of outgassing rates alone, it would appear that a surface treatment consisting of

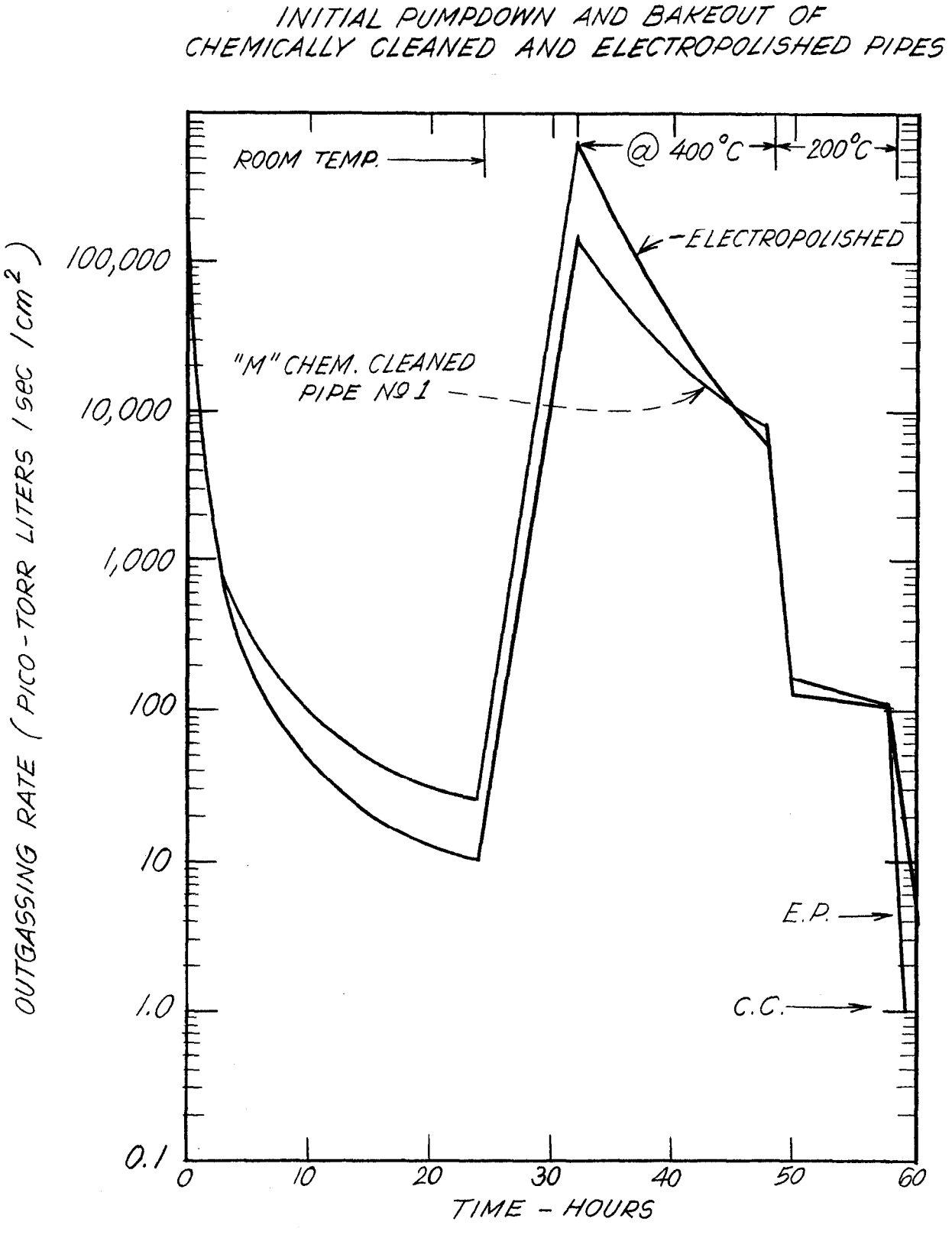

INITIAL PUMPDOWN AND BAKEOUT OF
CHEMICALLY CLEANED AND ELECTROPOLISHED PIPES
ROOM TEMP.
@ 400°C
200°C
ELECTROPOLISHED
"M" CHEM. CLEANED
PIPE NO 1
E.P.
C.C.
OUTGASSING RATE (PICO-TORR LITERS / SEC / cm²)
100,000
10,000
1,000
100
10
1.0
0.1
0
10
20
30
40
50
60
TIME - HOURS

FIGURE 2

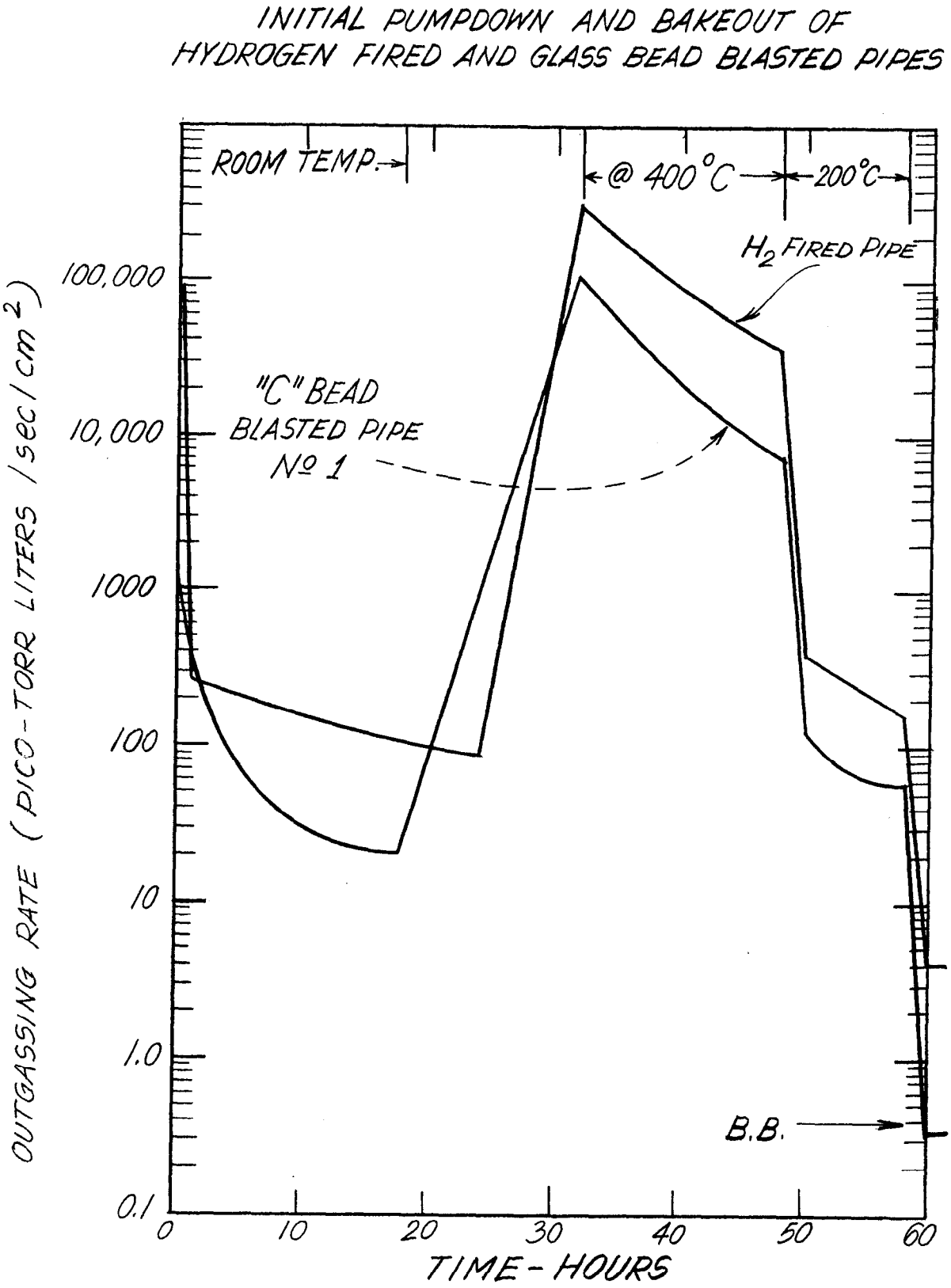

INITIAL PUMPDOWN AND BAKEOUT OF
HYDROGEN FIRED AND GLASS BEAD BLASTED PIPES
ROOM TEMP.
@ 400°C
200°C
H2 FIRED PIPE
"C" BEAD
BLASTED PIPE
Nº 1
B.B.
OUTGASSING RATE (PICO-TORR LITERS /SEC/CM2)
100,000
10,000
1000
100
10
1.0
0.1
0
10
20
30
40
50
60
TIME-HOURS

FIGURE 3

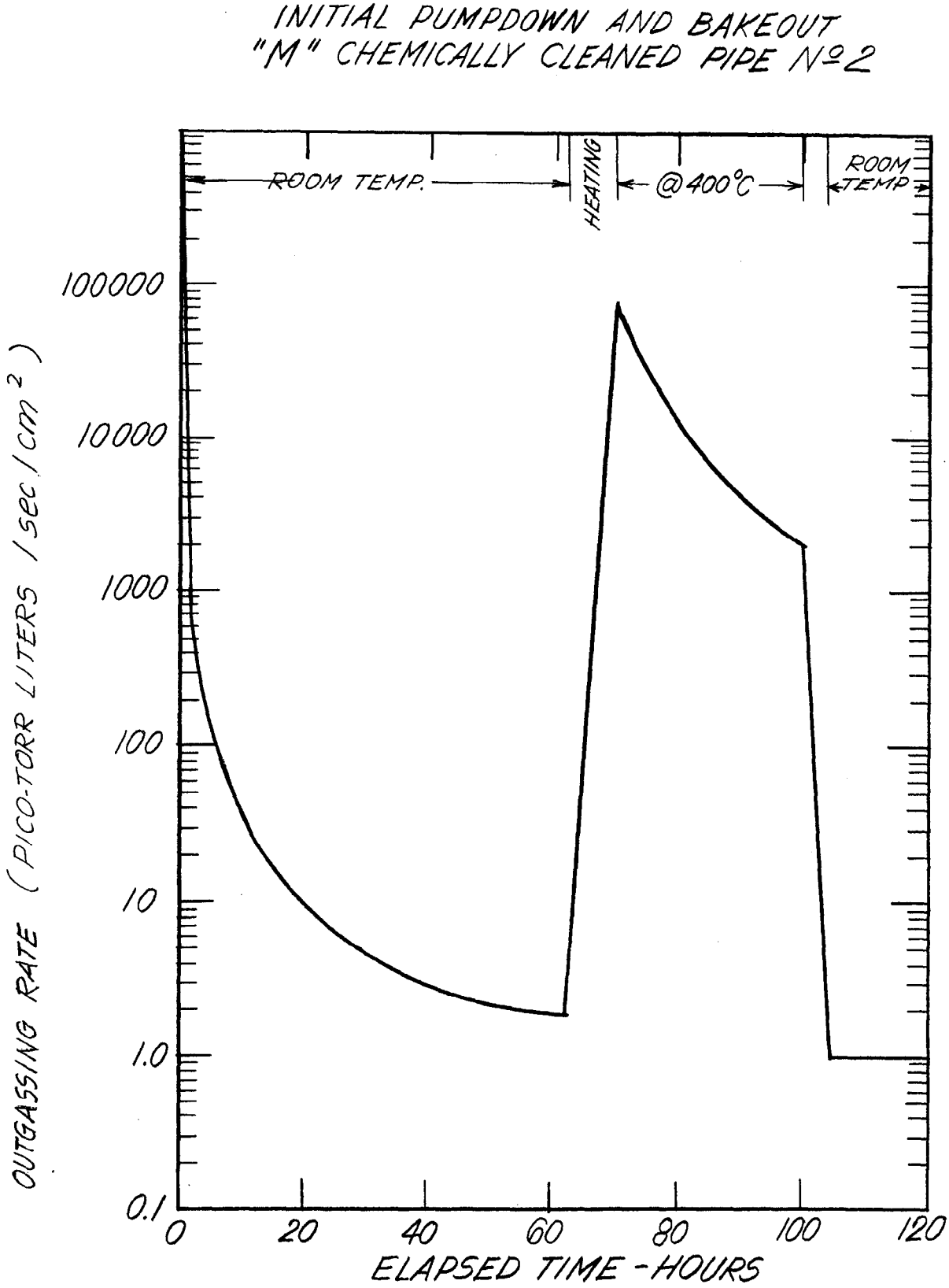

INITIAL PUMPDOWN AND BAKEOUT
"M" CHEMICALLY CLEANED PIPE Nº2
ROOM TEMP.
HEATING
@400°C
ROOM TEMP
100000
10000
1000
100
10
1.0
0.1
OUTGASSING RATE (PICO-TORR LITERS / SEC / CM²)
0
20
40
60
80
100
120
ELAPSED TIME - HOURS

FIGURE 4

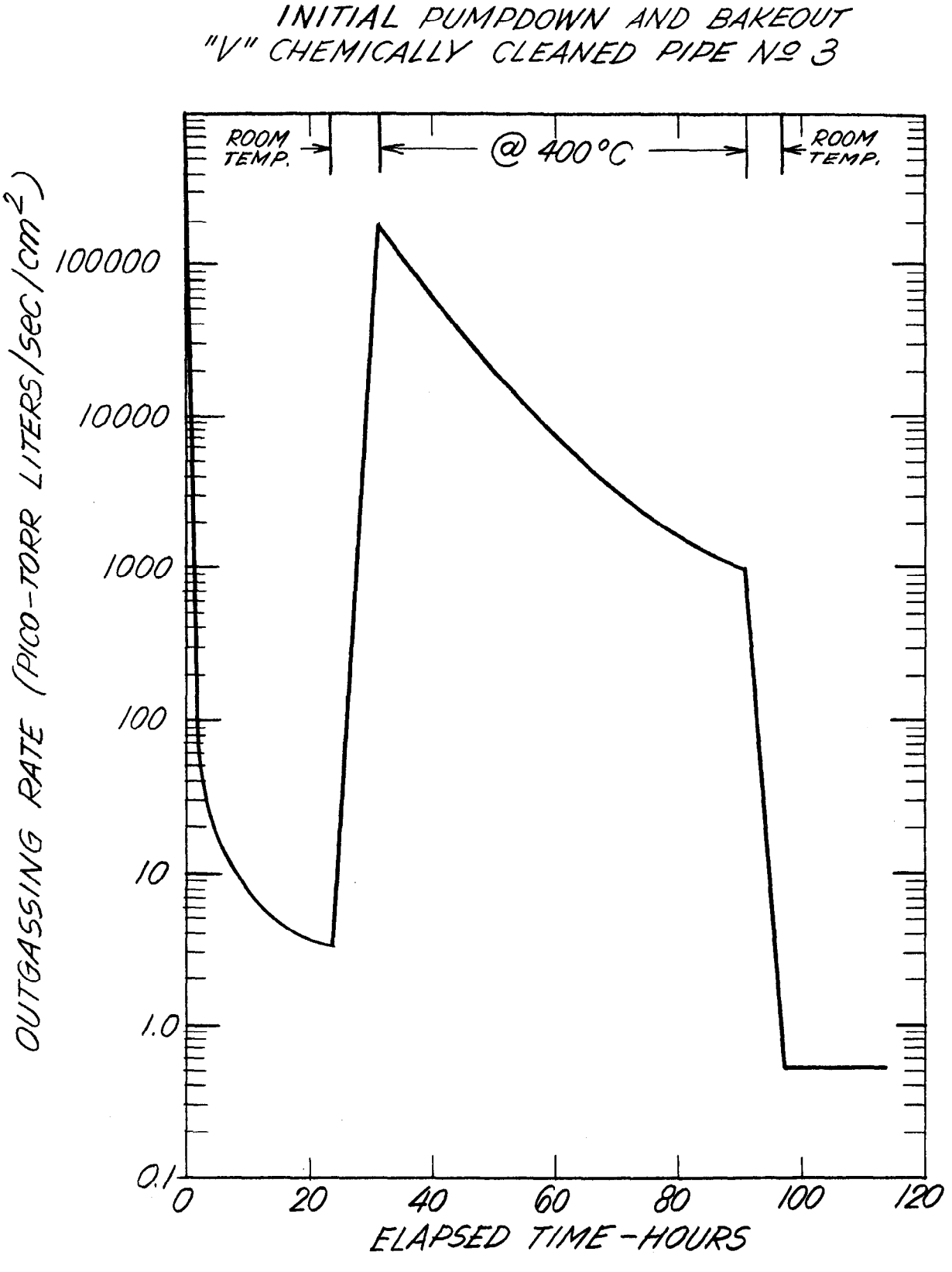

INITIAL PUMPDOWN AND BAKEOUT
"V" CHEMICALLY CLEANED PIPE Nº 3
ROOM TEMP.
@ 400°C
ROOM TEMP.
OUTGASSING RATE (PICO-TORR LITERS/SEC/CM²)
100000
10000
1000
100
10
1.0
0.1
0
20
40
60
80
100
120
ELAPSED TIME-HOURS

FIGURE 5

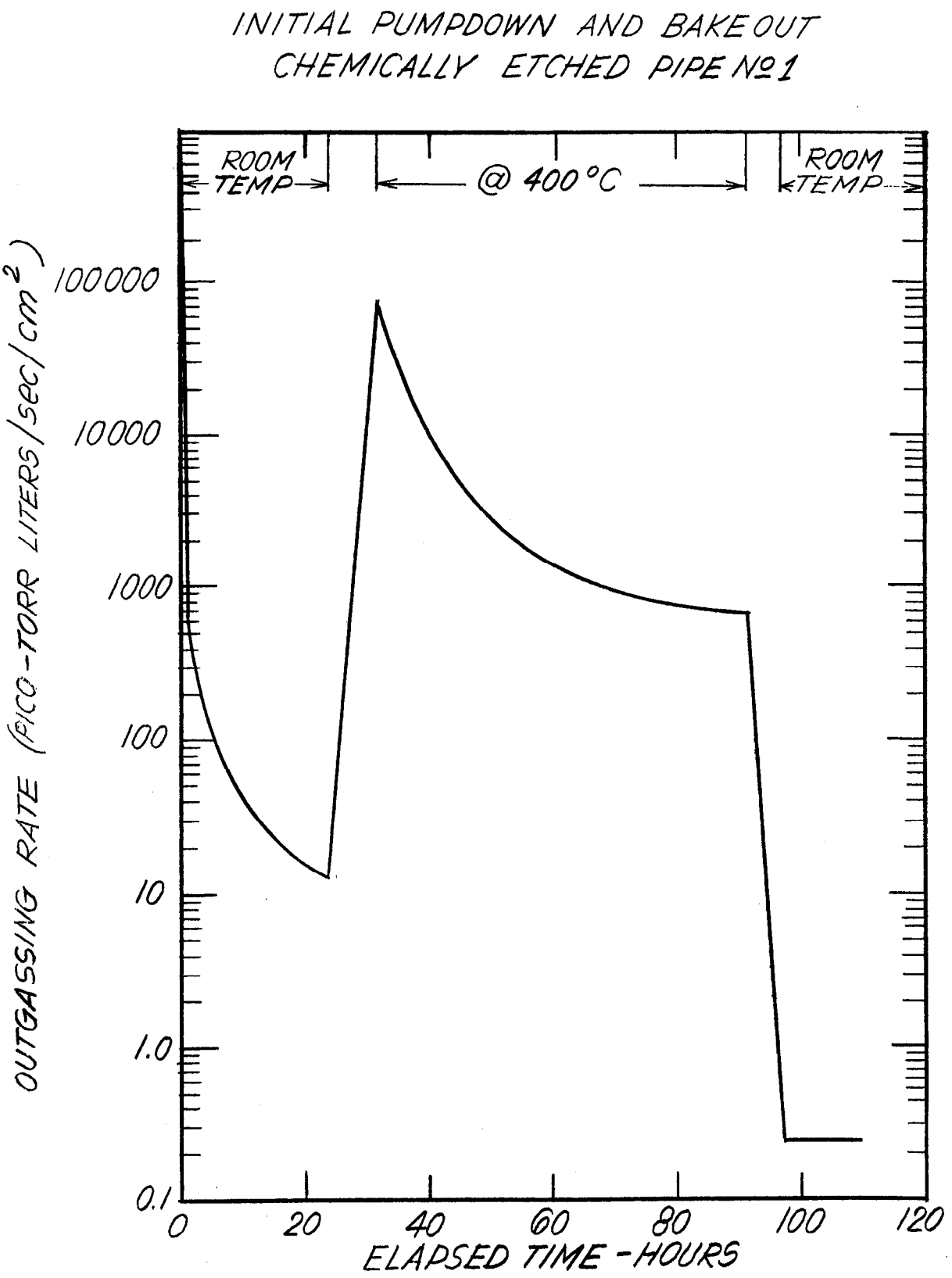

INITIAL PUMPDOWN AND BAKEOUT
CHEMICALLY ETCHED PIPE Nº1
ROOM TEMP
@ 400 °C
ROOM TEMP
OUTGASSING RATE (PICO-TORR LITERS/SEC/CM²)
100000
10000
1000
100
10
1.0
0.1
0
20
40
60
80
100
120
ELAPSED TIME - HOURS

FIGURE 6

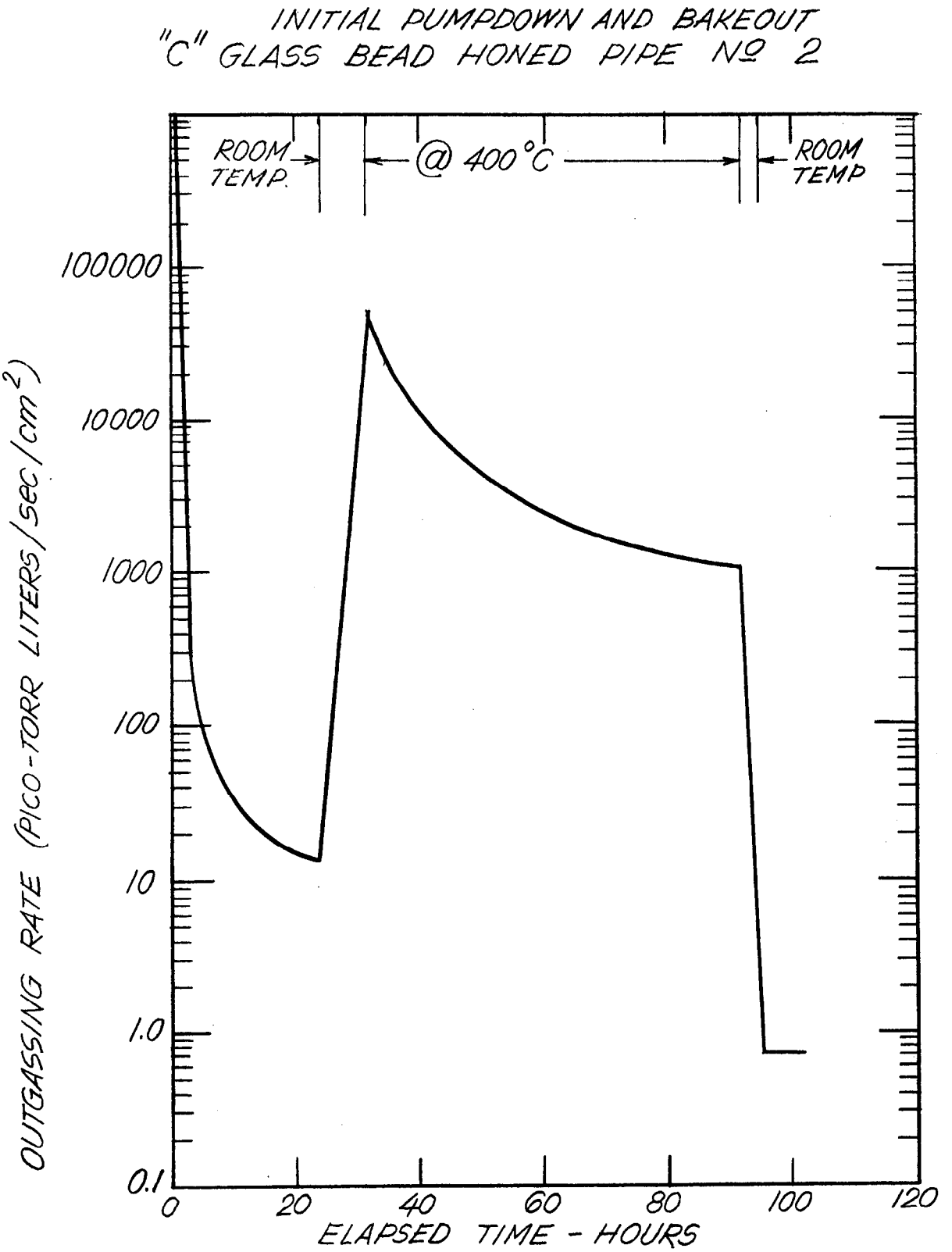

INITIAL PUMPDOWN AND BAKEOUT
"C" GLASS BEAD HONED PIPE Nº 2
ROOM TEMP.
@ 400°C
ROOM TEMP
100000
10000
1000
100
10
1.0
0.1
OUTGASSING RATE (PICO-TORR LITERS/SEC/CM²)
0
20
40
60
80
100
120
ELAPSED TIME - HOURS

FIGURE 7

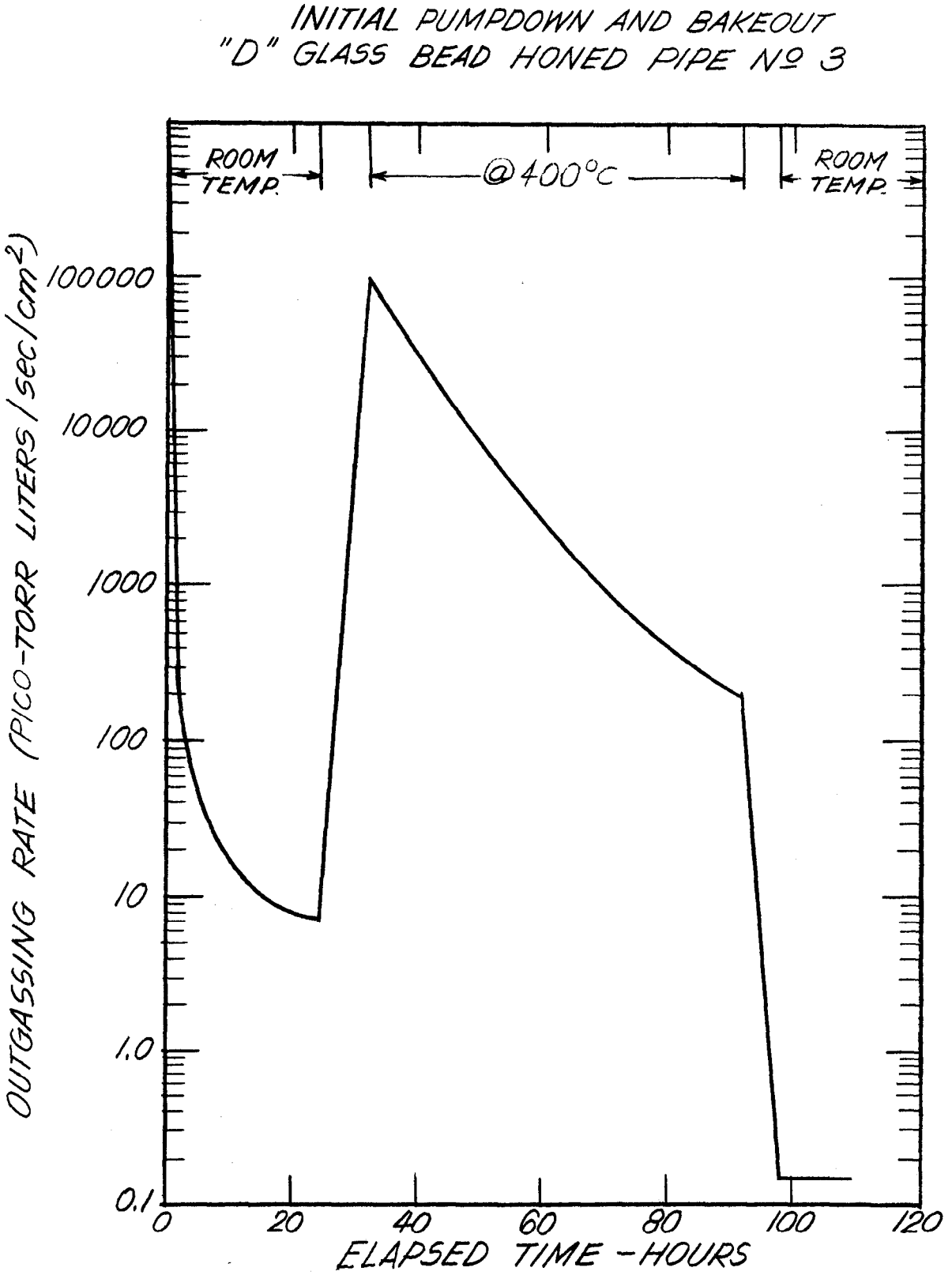

INITIAL PUMPDOWN AND BAKEOUT
"D" GLASS BEAD HONED PIPE Nº 3
ROOM TEMP.
@ 400°C
ROOM TEMP.
OUTGASSING RATE (PICO-TORR LITERS / sec / cm²)
100000
10000
1000
100
10
1.0
0.1
0
20
40
60
80
100
120
ELAPSED TIME - HOURS

FIGURE 8

degreasing and glass bead blasting would be most desirable. However, when we examine the pumpdown behavior following air and nitrogen exposures, a different picture emerges.

Outgassing rate data for all pipes tested are summarized in Table IV.

Figures 9 through 15 are log-log plots of the system total pressure versus time for eight of the nine pipes tested, during initial pumpdown and pumpdown following air or nitrogen exposures after bakeout. Figure 9 depicts the pumpdown performance of the hydrogen-fired pipe. After baking, this pipe was exposed to nitrogen taken directly from a cylinder of high purity dry nitrogen. Residual gas analysis during subsequent pumpdown showed the presence of hydrocarbons. It was concluded that the observed hydrocarbons were coming from the cylinder or connecting lines, and it was therefore decided to repeat the exposure with nitrogen passed through a trap at liquid nitrogen temperature. The second nitrogen pumpdown showed much fewer hydrocarbons; all of the subsequent nitrogen exposures on the other test pipes were made using nitrogen taken from boiling liquid nitrogen.

Figure 10 shows the pumpdown performance of the electropolished pipe. It can be seen that exposure time has little effect on subsequent pumpdown behavior once the surfaces have been baked.

Figures 11, 12 and 13 compare the initial pumpdowns of the chemically cleaned and etched pipes with pumpdown behavior after air and nitrogen exposures. It is significant that little difference in pumpdown time was noted for nitrogen exposures from 1 to 750 hours, and air exposures of 1 and 200 hours.

Figures 14 and 15 depict the pumpdown behavior of the three glass bead blasted pipes. It will be noted that the pumpdown curves for the bead blasted surfaces exposed after bakeout cross the initial pumpdown curve at some low pressure. The reason for this behavior is not fully understood, but it is probably related to the fact that the bead blasting operation produced a martensitically transformed surface. Metallographic examination showed the transformation thickness to be limited by the grain size, each grain at the surface being transformed from the F.C.C. austenite phase to the B.C.C. martensite phase. In the pipes examined the grain size and consequent depth of the martensite phase was between 25μ and 250μ.

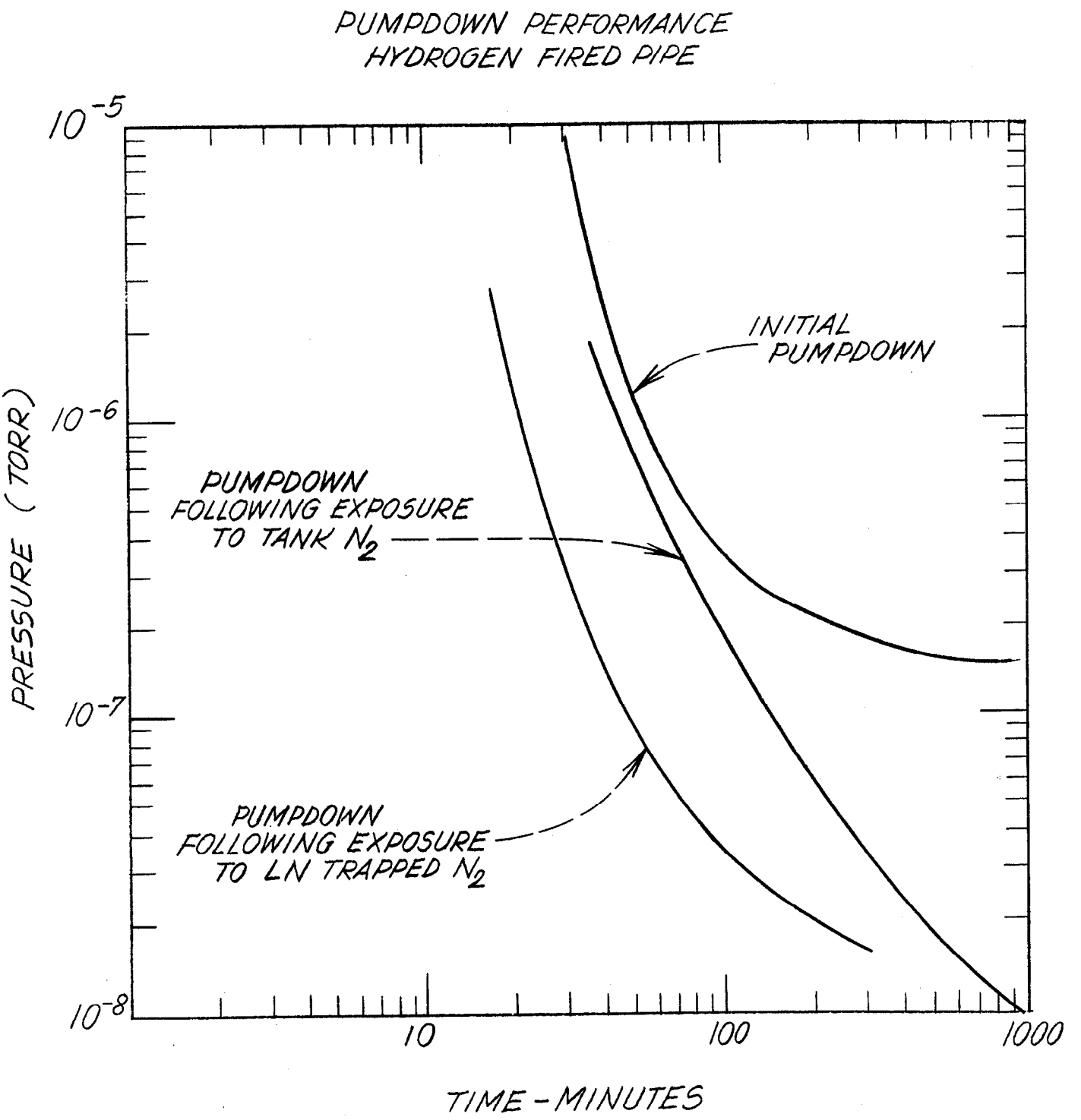

PUMPDOWN PERFORMANCE
HYDROGEN FIRED PIPE
$10^{-5}$
$10^{-6}$
$10^{-7}$
$10^{-8}$
PRESSURE (TORR)
INITIAL PUMPDOWN
PUMPDOWN FOLLOWING EXPOSURE TO TANK $N_2$
PUMPDOWN FOLLOWING EXPOSURE TO LN TRAPPED $N_2$
10
100
1000
TIME - MINUTES

FIGURE 9

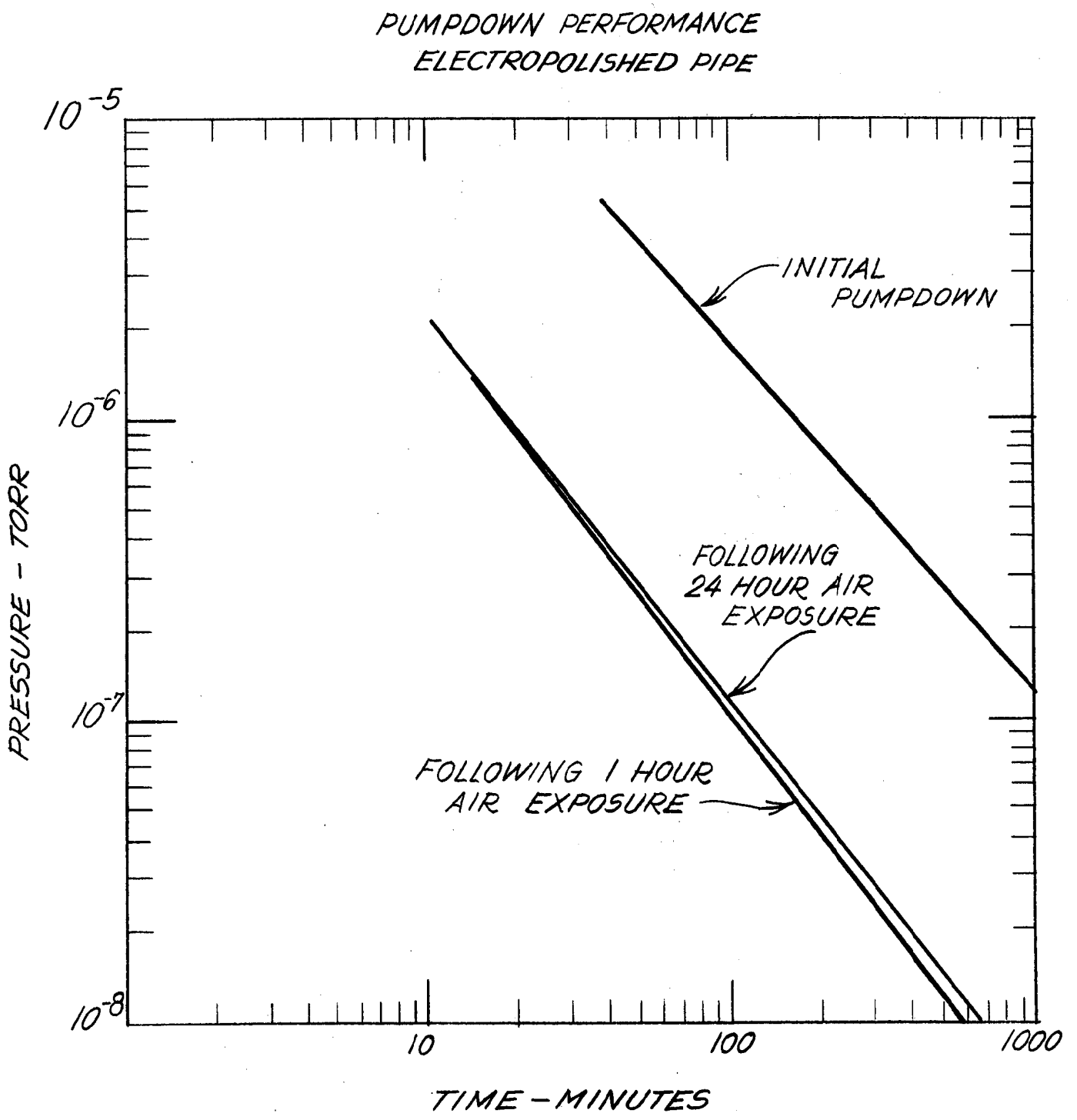

PUMPDOWN PERFORMANCE
ELECTROPOLISHED PIPE
$10^{-5}$
$10^{-6}$
$10^{-7}$
$10^{-8}$
PRESSURE - TORR
INITIAL PUMPDOWN
FOLLOWING 24 HOUR AIR EXPOSURE
FOLLOWING 1 HOUR AIR EXPOSURE
10
100
1000
TIME - MINUTES

FIGURE 10

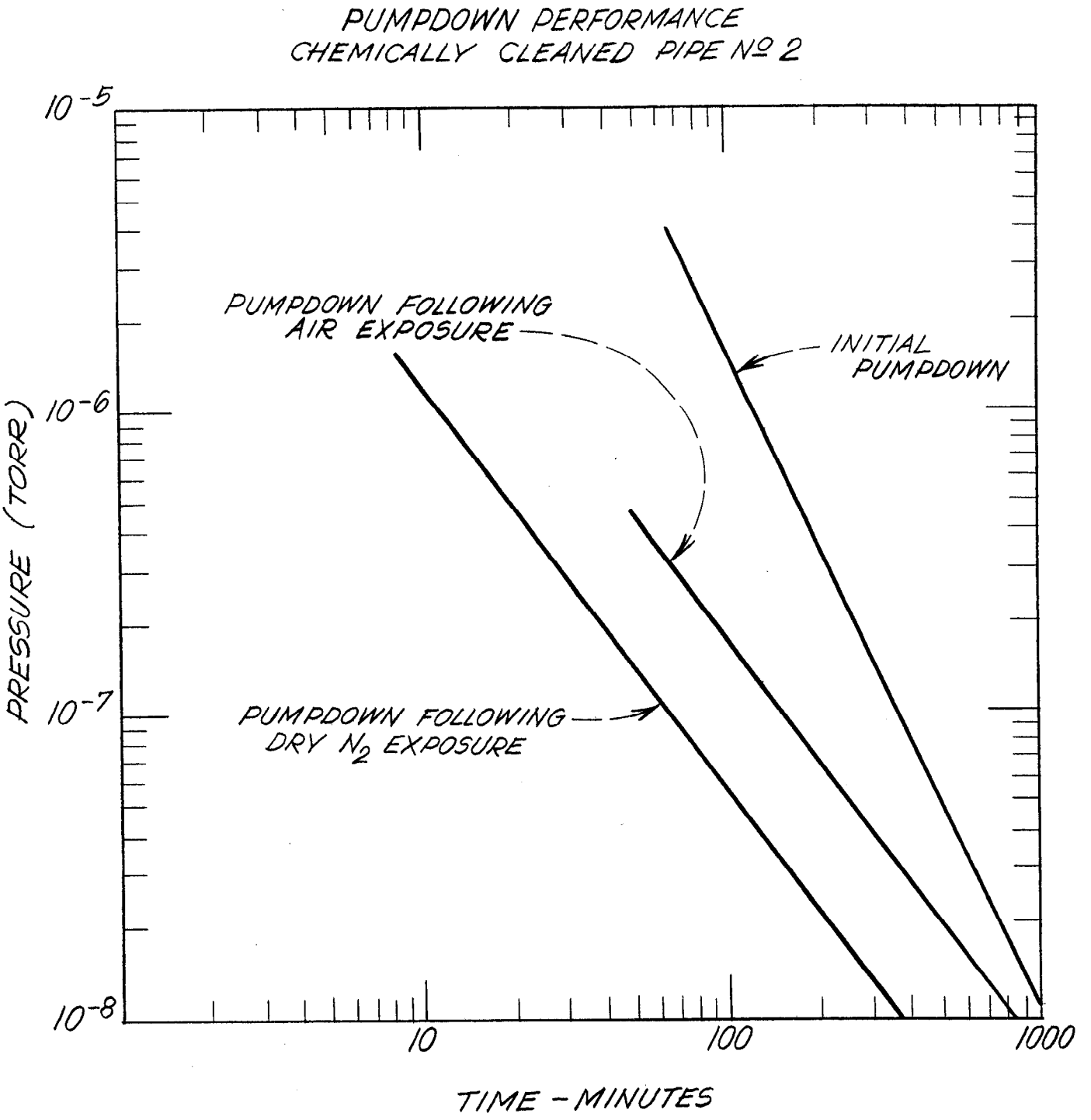

PUMPDOWN PERFORMANCE
CHEMICALLY CLEANED PIPE Nº 2
PRESSURE (TORR)
10⁻⁵
10⁻⁶
10⁻⁷
10⁻⁸
PUMPDOWN FOLLOWING AIR EXPOSURE
INITIAL PUMPDOWN
PUMPDOWN FOLLOWING DRY N2 EXPOSURE
10
100
1000
TIME - MINUTES

FIGURE 11

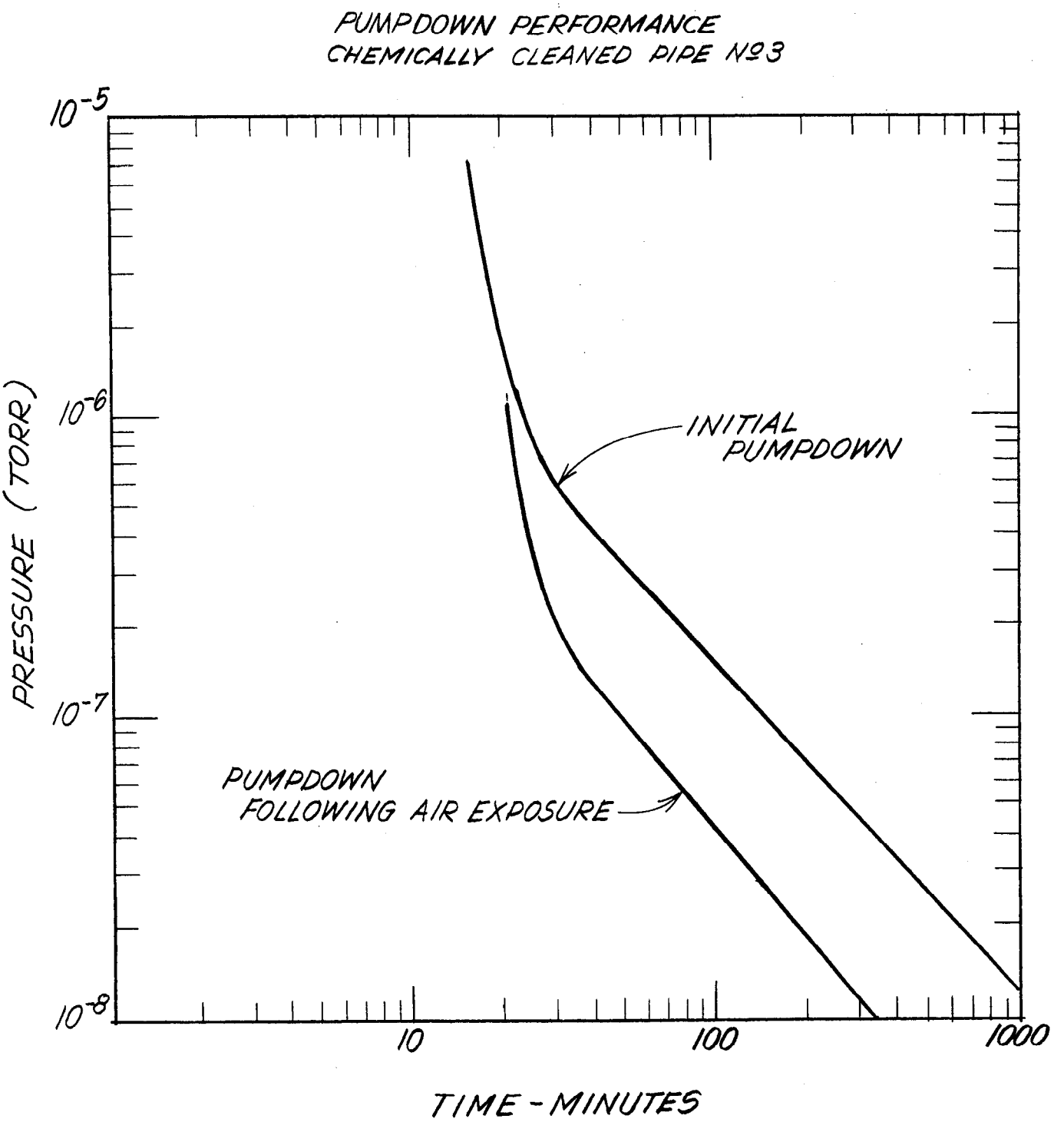

PUMPDOWN PERFORMANCE
CHEMICALLY CLEANED PIPE Nº3
$10^{-5}$
$10^{-6}$
$10^{-7}$
$10^{-8}$
PRESSURE (TORR)
INITIAL PUMPDOWN
PUMPDOWN FOLLOWING AIR EXPOSURE
10
100
1000
TIME - MINUTES

FIGURE 12

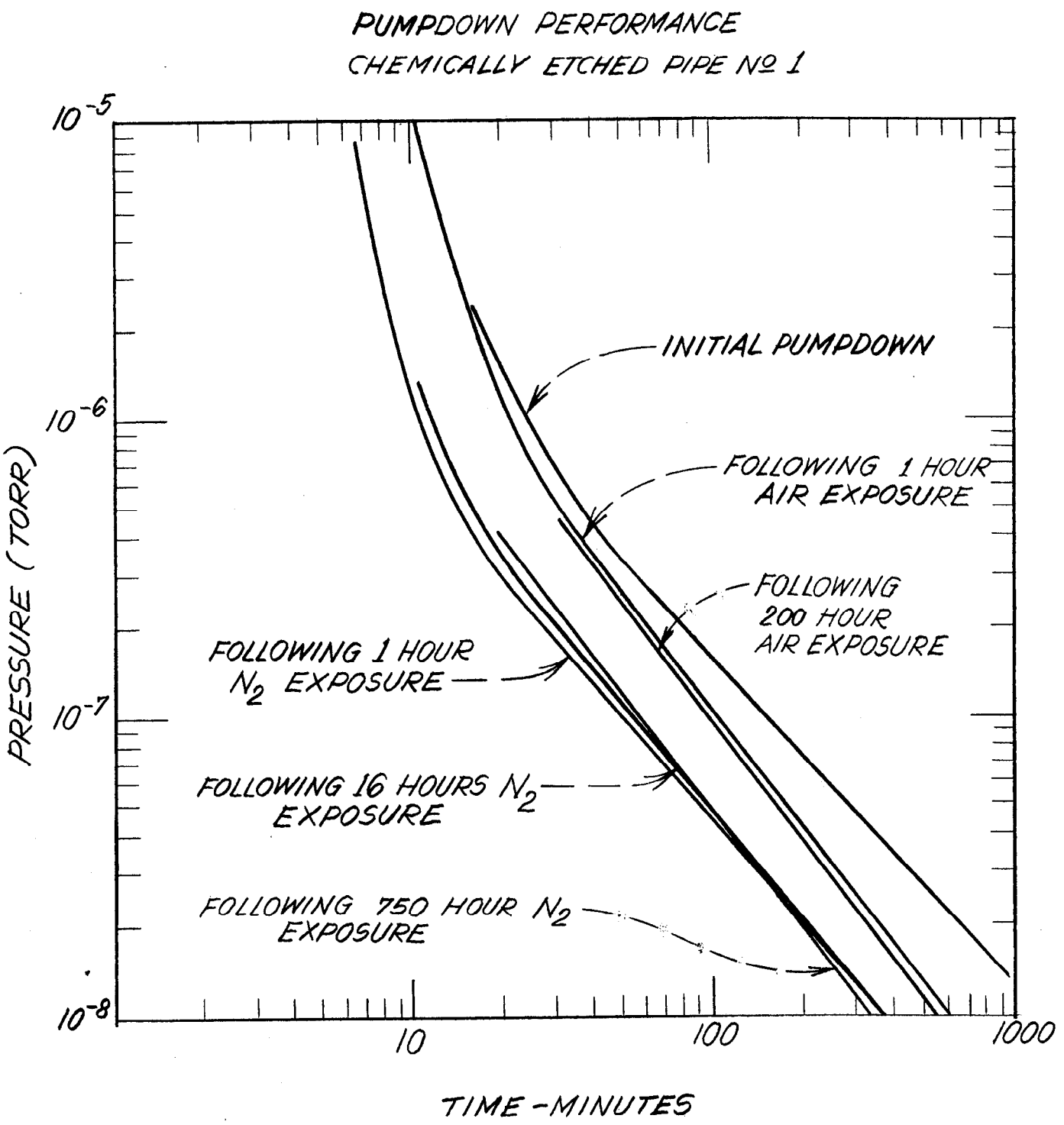

PUMPDOWN PERFORMANCE
CHEMICALLY ETCHED PIPE Nº 1
PRESSURE (TORR)
$10^{-5}$
$10^{-6}$
$10^{-7}$
$10^{-8}$
INITIAL PUMPDOWN
FOLLOWING 1 HOUR AIR EXPOSURE
FOLLOWING 200 HOUR AIR EXPOSURE
FOLLOWING 1 HOUR $N_2$ EXPOSURE
FOLLOWING 16 HOURS $N_2$ EXPOSURE
FOLLOWING 750 HOUR $N_2$ EXPOSURE
10
100
1000
TIME-MINUTES

FIGURE 13

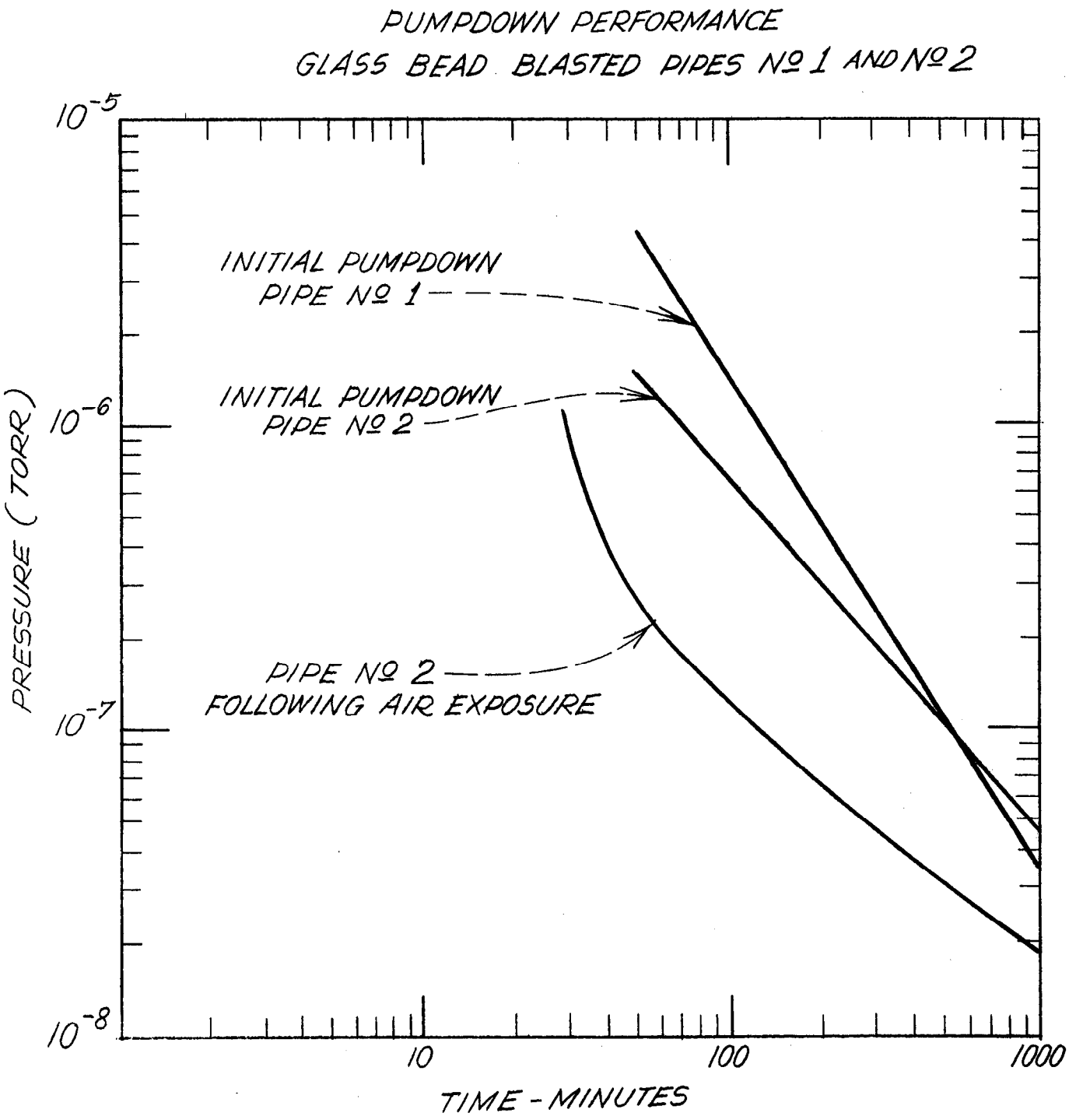

PUMPDOWN PERFORMANCE
GLASS BEAD BLASTED PIPES Nº 1 AND Nº 2
INITIAL PUMPDOWN
PIPE Nº 1
INITIAL PUMPDOWN
PIPE Nº 2
PIPE Nº 2
FOLLOWING AIR EXPOSURE
PRESSURE ( TORR )
$10^{-5}$
$10^{-6}$
$10^{-7}$
$10^{-8}$
10
100
1000
TIME - MINUTES

FIGURE 14

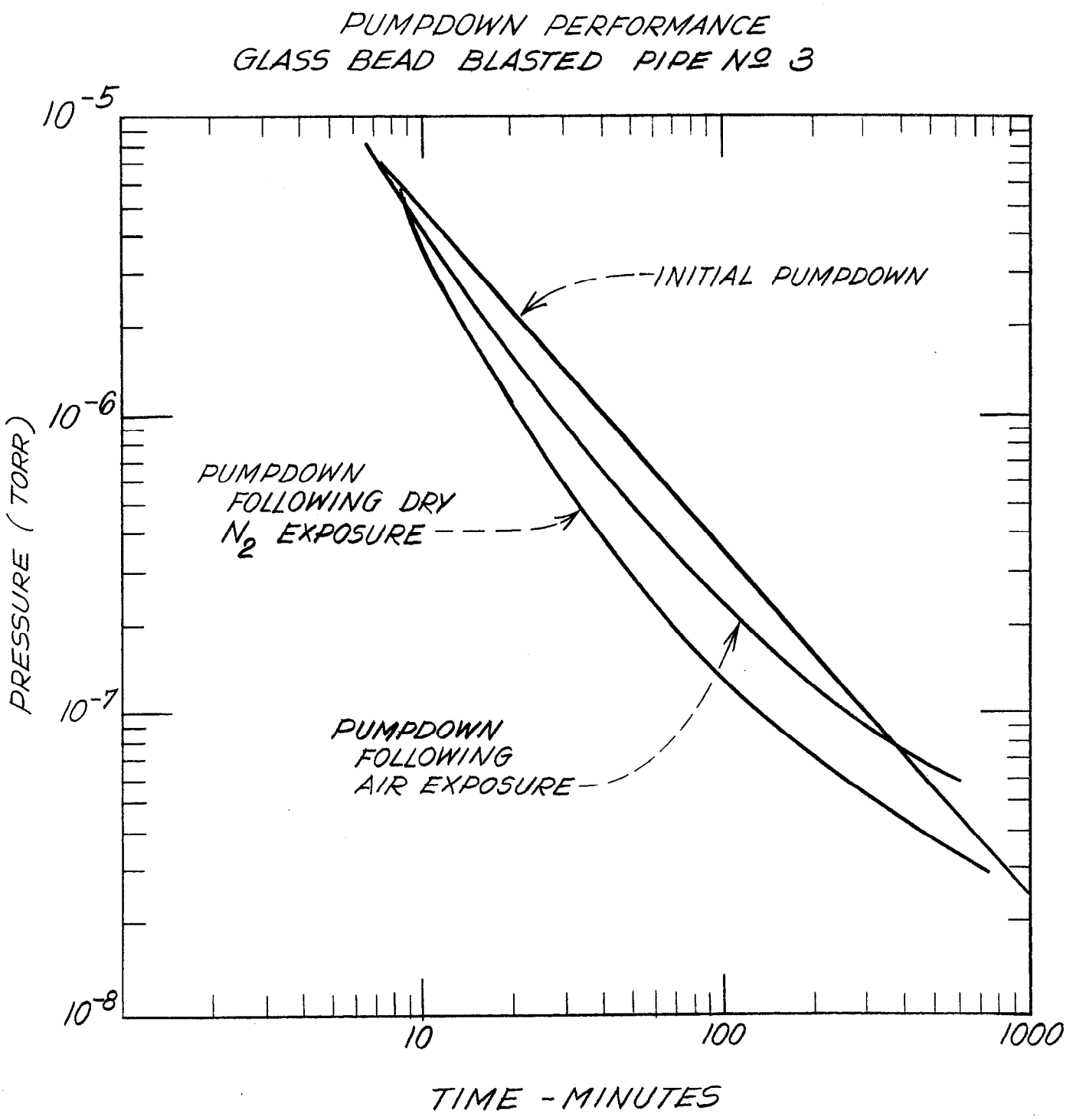

PUMPDOWN PERFORMANCE
GLASS BEAD BLASTED PIPE Nº 3
$10^{-5}$
$10^{-6}$
$10^{-7}$
$10^{-8}$
PRESSURE ( TORR )
INITIAL PUMPDOWN
PUMPDOWN FOLLOWING DRY $N_2$ EXPOSURE
PUMPDOWN FOLLOWING AIR EXPOSURE
10
100
1000
TIME - MINUTES

FIGURE 15

The equilibrium solubility of carbon in the body-centered cubic phase is 0.02 percent, whereas the carbon content of type 304 stainless steel is nominally 0.08 percent as was the case in point. As a result of the diffusionless transformation, the martensite phase is supersaturated with carbon. X-ray diffraction examination of the transformed surfaces before and after vacuum bakeout at 400°C showed much less distortion and tetragonality of the crystals on the surface that was baked. This suggests that some of the extra carbon left the lattice during the bakeout. Residual gas analysis made during the early runs showed that much more mass 44($CO_2$ or $C_3H_8$) was released from the glass bead treated pipe than from the others.

Figures 16 and 17 show the results of a second and third bakeout following the initial bake and air exposure. It is apparent that little is gained by baking at temperatures below the original bakeout temperature.

Figure 18 depicts the results of extended pumping at room temperature following air exposure of a baked pipe. In this case an outgassing rate as low as that attained after bakeout was achieved in four days of pumping after air exposure.

The results reported for all the room temperature outgassing rates before and after bakeout have been adjusted to 20°C. It was found that variations in ambient temperature had a significant effect on the outgassing rates.

Some typical temperature coefficients for baked pipes following air or nitrogen exposure and subsequent pumpdown were as follows:

| Pipe | Temperature Coefficient (15 - 35°C) of Outgassing (pico-torr liters/sec/cm²/°C) |
|---|---|
| Glass bead blasted #2 | 0.05 |
| Chemically cleaned #2 | 0.07 |
| Etched #1 | 0.05 |

These numbers are based on a very limited number of measurements and are reported only to show the importance of temperature variations on outgassing rates.

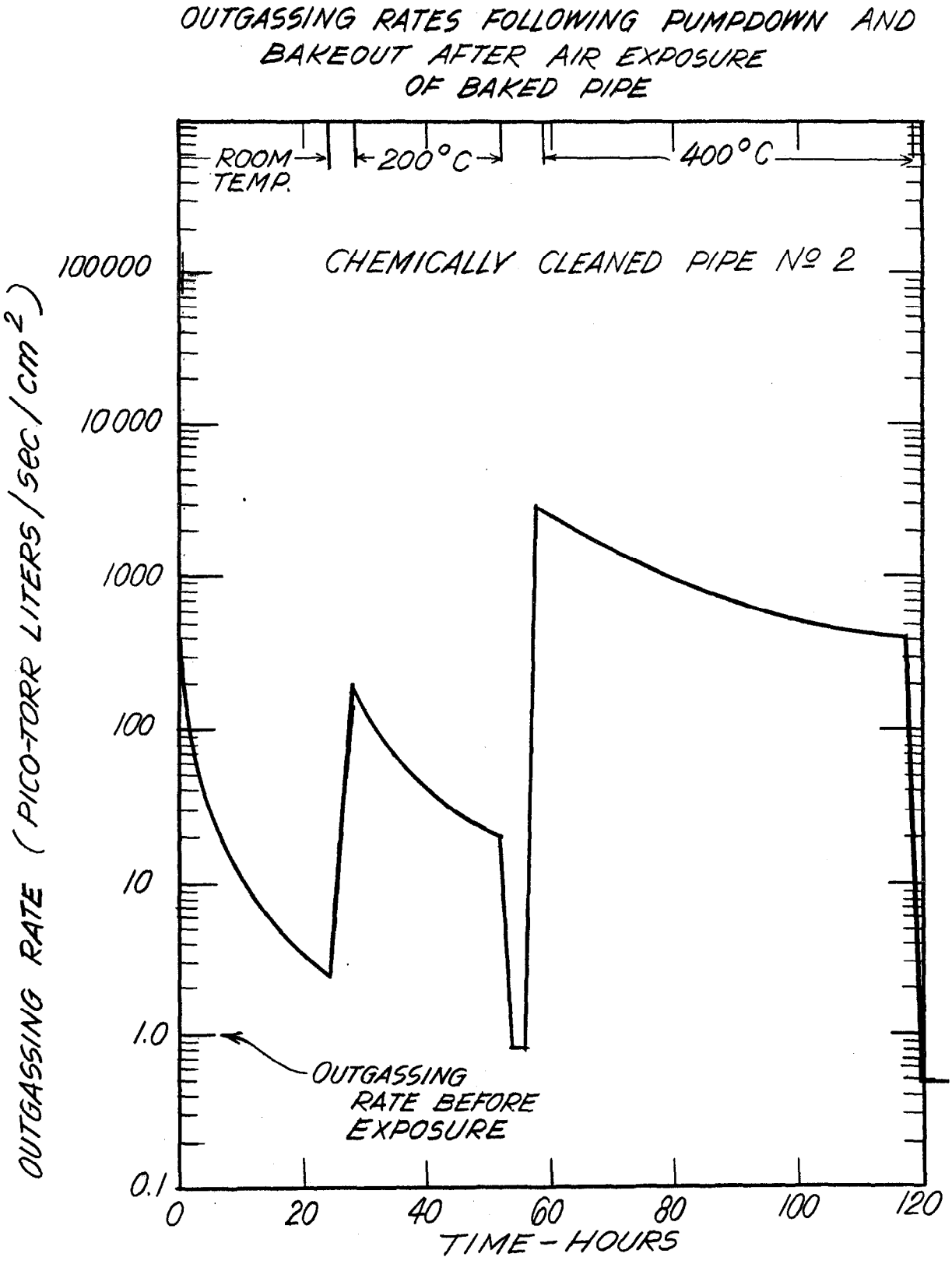

OUTGASSING RATES FOLLOWING PUMPDOWN AND BAKEOUT AFTER AIR EXPOSURE OF BAKED PIPE
ROOM TEMP.
200°C
400°C
CHEMICALLY CLEANED PIPE Nº 2
OUTGASSING RATE (PICO-TORR LITERS/SEC/CM²)
100000
10000
1000
100
10
1.0
0.1
OUTGASSING RATE BEFORE EXPOSURE
0
20
40
60
80
100
120
TIME-HOURS

FIGURE 16

OUTGASSING RATE FOLLOWING PUMPDOWN AND
200 °C BAKE AFTER AIR EXPOSURE OF BAKED PIPE

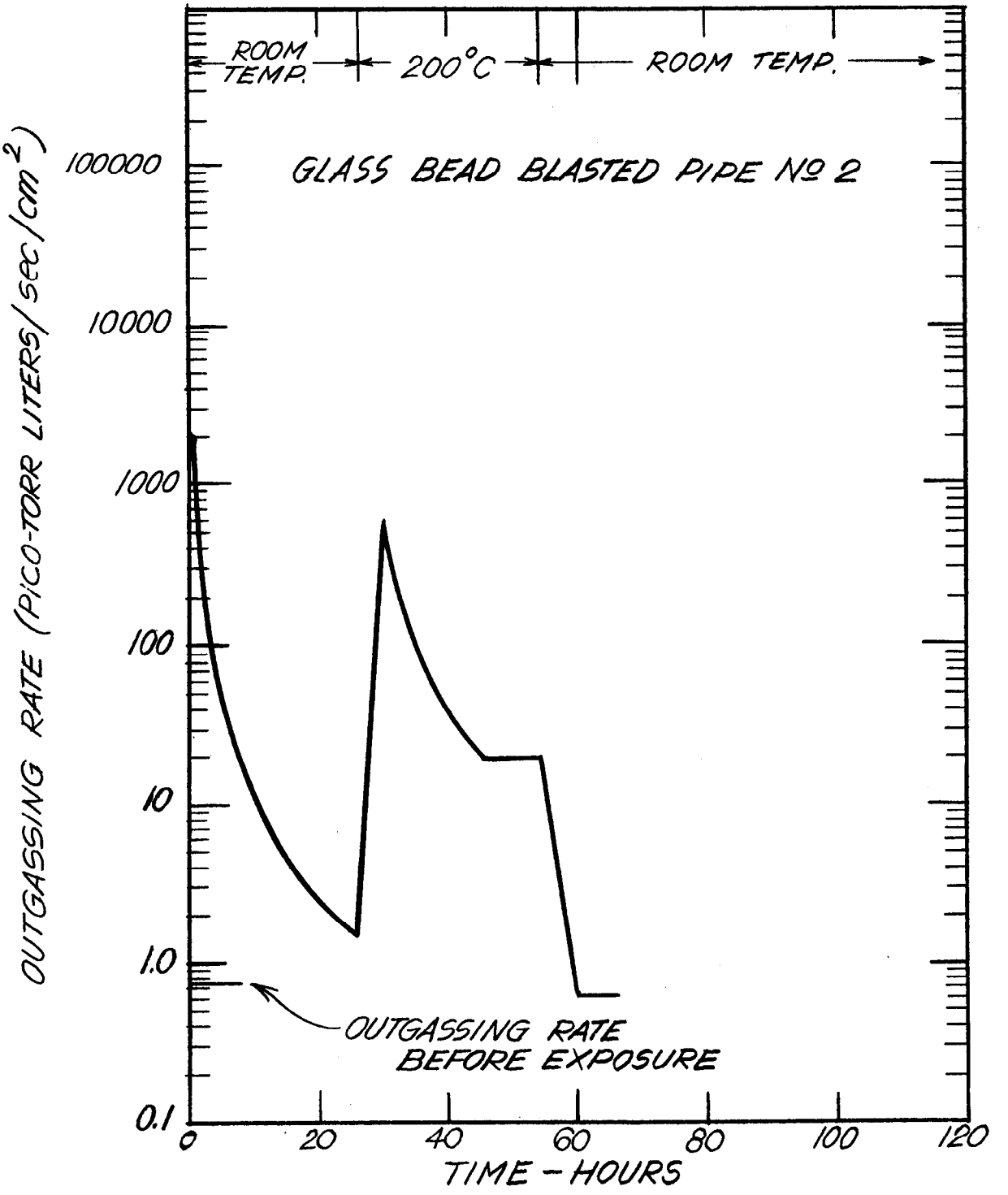



FIGURE 17

ROOM TEMPERATURE OUTGASSING
RATE FOLLOWING AIR EXPOSURE OF BAKED PIPE

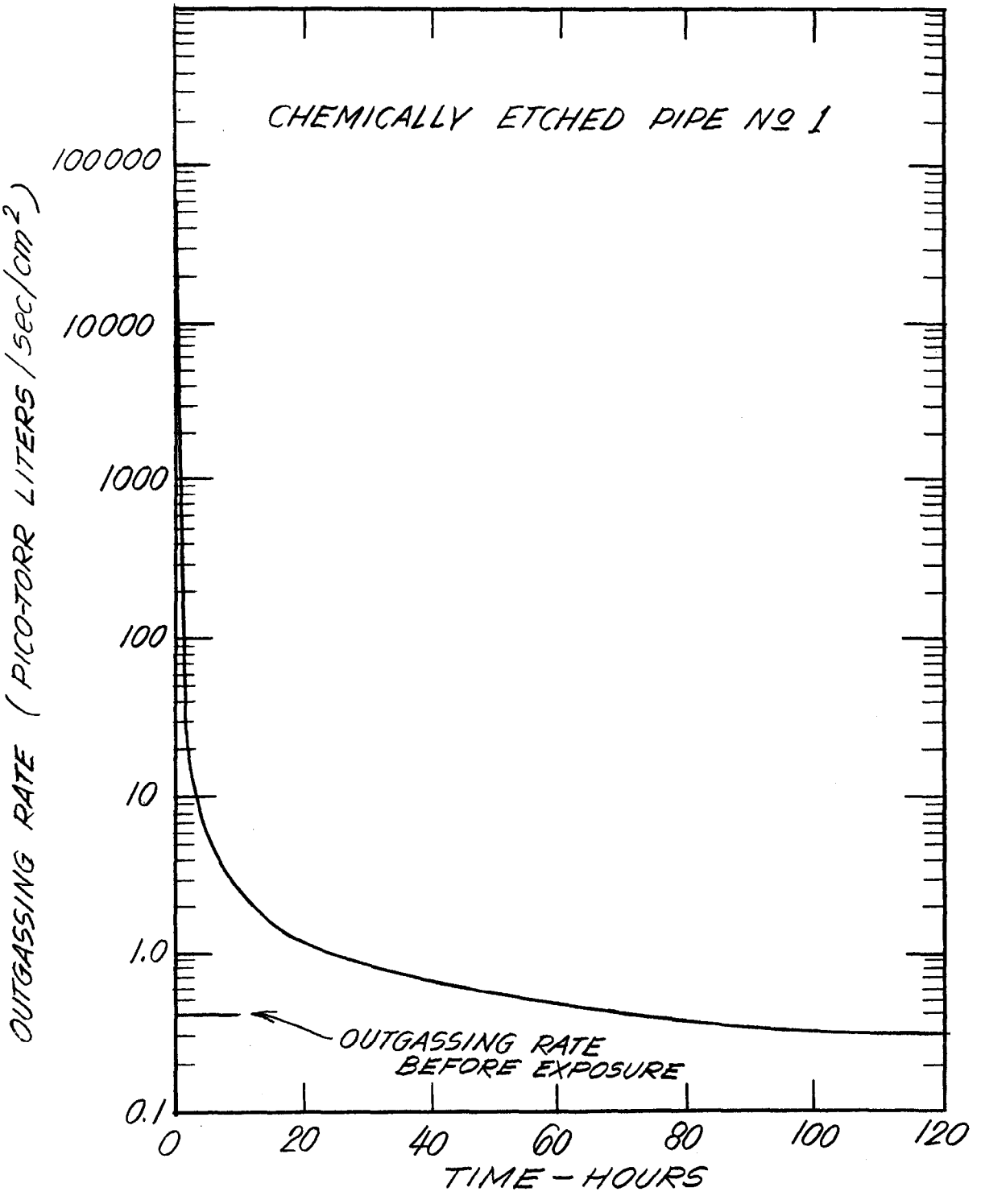



FIGURE 18

## V. CONCLUSIONS

Returning to the original five questions, the following answers are offered.

1. The lowest relative outgassing rates can be attained with bead-blasted surfaces. However, chemically cleaned surfaces, especially if some surface is removed, attain nearly as low outgassing rates and have an advantage in cleaning up faster after air or nitrogen exposure.

2. Baking chemically cleaned surfaces at $400^{\circ}C$ for times in excess of 48 hours will yield room temperature outgassing rates below 1 pico-torr liter/sec/$cm^2$.

3. If baked piping is exposed to nitrogen or even moist air, recovery to outgassing rates near those attained after baking can be expected within four days of pumping at room temperature.

4. There is a definite advantage in exposing baked piping to nitrogen rather than to air. However, clean air exposures (that is, air without entrained particulate matter or insects) should not be catastrophic. The results of the tests reported here indicate an extension of the pumpdown time by at most a factor of three for air over nitrogen.

5. There is little evidence that time of exposure to clean air or nitrogen has any significant effect on subsequent pumpdown behavior.

## ACKNOWLEDGMENT

Grateful acknowledgment is made to J. R. Balch, who performed most of the tedious assembly and disassembly operations, in addition to making measurements at odd times of the day or night as required.

The following people contributed many helpful suggestions and ideas during the course of the work reported: Dr. E. L. Garwin and J. A. Pope of SLAC, Ray Flaig of Vacu-Blast Company, Inc., John Cape of Varian Associates, and W. V. Cummings and S. J. Armijo of G. E. Vallecitos Atomic Laboratory.

TABLE I

SURFACE TREATMENTS

Hydrogen Firing

1. Vapor degrease
2. Hydrogen fired for 30 minutes at 1000°C - 80°F dewpoint

Electropolish

1. Vapor degrease
2. Alkaline clean
3. Tap water rinse
4. Electropolish (in Battelle solution for 300 series SS) for 25 minutes at 95°C, 7 volts - 0.1 amp/cm 2, copper anode
5. Distilled water rinse
6. Methanol rinse

Chemical Etch

1. Vapor degrease
2. Alkaline clean
3. Tap water rinse
4. Etch 2 hours - room temperature. 1/3 $HNO_3$, 1/3 HF, 1/3 $H_2O$ (This etch removed approximately 0.0005 inch from the surface.)
5. Tap water rinse
6. Cold distilled water rinse
7. Hot distilled water rinse (5 minutes)
8. Dry nitrogen blast

Chemical Cleaning "M"

1. Vapor degrease
2. Alkaline clean
3. Tap water rinse
4. Acid clean; 7-1/2 $H_2O$, 2-1/2 $HC\ell$, 1/2 $HNO_3$; 2 to 3 minutes, 70°C
5. Tap water rinse
6. Cold distilled water rinse
7. Cold distilled water rinse
8. Methanol rinse
9. Dry in oven at 140°C

Chemical Cleaning "V," Pipe #3, Material "B"

1. Vapor degrease
2. Alkaline clean
3. Hot tap water rinse
4. Acid clean; 20 to 25% $HNO_3$, 2 to 3% HF; 10 minutes, 50° to 60°C
5. Cold tap water rinse
6. Hydro wash (80 lbs. air + $H_2O$ mix)
7. Cold tap water rinse
8. Deionized water rinse 80°C
9. Deionized water rinse 95°C
10. Clean air blow-off

Glass Bead Blasting "C"

1. Chemically cleaned (recipe "M")
2. Internal surfaces blasted with glass beads* 10 to 53μ in diameter

Glass Bead Blasting "D," Pipe #3, Material "B"

1. Vapor degreased
2. Blasted with glass beads 10 to 53μ in diameter

TABLE II

CHEMICAL ANALYSIS OF STAINLESS STEEL PIPES

| | "A" | "B" |
|---|---|---|
| Chromium | 18.8 | 18.7 |
| Manganese | 1.40 | 1.10 |
| Molybdenum | 0.35 | 0.36 |
| Nickel | 9.3 | 9.6 |
| Silicon | 1.10 | 0.95 |
| Carbon | 0.08 | 0.067 |
| Phosphorus | 0.015 | 0.010 |
| Sulfur | 0.003 | 0.010 |

*Glas-shot MS-XL available from Micro Beads, Inc., Jackson, Mississippi.

TABLE III

RESIDUAL GAS ANALYSIS

| Condition | Principal Residual Gases (in order of concentration) Surface Treatment | | | |
|---|---|---|---|---|
| | $H_2$ fired | Electropolished | Chemically cleaned | Glass bead honed |
| Following room temperature | $H_2$, CO, $H_2O$, $CO_2$ | $H_2$, CO, $CO_2$, $H_2O$ | CO, $H_2O$, $CO_2$ | $H_2O$, CO, $CO_2$ |
| Pumpdown for 24 hours | $CH_4$, $C_3H_8$, $C_2H_6$ | $N_2$, $CH_4$, $C_2H_6$ $C_3H_8$ | $H_2$, $CH_4$, $C_3H_8$ | $CH_4$, $H_2$, $C_3H_8$ $C_2H_6$ |
| During 400°C bakeout | CO, $CO_2$, $H_2O$ $H_2$, $CH_4$, $C_3H_6$ $C_2H_6$ | CO, $H_2O$, $CH_4$ $C_3H_8 + C_4H_{10}$ $C_4H_{10}$ | $H_2$, CO, $H_2O$ Ar , $CH_4$ | $H_2$, $CO_2$, CO $H_2O$, $CH_4$, $C_3H_8$ $C_4H_{10}$ |
| Room temperature | CO, $H_2O$, $CO_2$ | CO, $H_2O$, $CO_2$ $C_4H_{10}$ | $H_2$, CO, $H_2O$ $CO_2$, $CH_4$ | $H_2$, CO, $H_2O$, $C_3H_8$ $CO_2$, $CH_4$ |

TABLE IV

OUTGASSING RATES

FOLLOWING AIR AND NITROGEN EXPOSURES

OF BAKED STAINLESS STEEL PIPES

| Surface Treatment | Outgassing Rate Before Exposure (pico-torr liters/sec/cm$^2$) | Room Temperature Exposure Conditions | Outgassing Rate Following Exposure After ≈24 Hours Pumping (pico-torr liters/sec/cm$^2$) |
|---|---|---|---|
| "A" Material | | | |
| $H_2$ fired | 5.3 | 1 hour tank $N_2$ | 23.3 |
| $H_2$ fired | 23.3 | 1 hour LN trapped $N_2$ | 18.2 |
| Electropolished | 3.2 | 1 hour air, dewpoint 13°C | 3.6 |
| Electropolished | 3.6 | 24 hours air, dewpoint 13°C | 3.2 |
| Chemically cleaned | 1.0 | 1 hour air, dewpoint 11°C | 2.4 |
| Chemically cleaned | 0.42 | 1 hour dry $N_2$ | 1.0 |
| Bead blasted | 0.78 | 1 hour air, dewpoint 13°C | 1.8 |
| Chemically etched | 0.25 | 1 hour dry $N_2$ | 0.51 |
| Chemically etched | 0.51 | 1 hour air, dewpoint 14°C | 1.3 |
| Chemically etched | 0.29 | 16 hours dry $N_2$ | 0.4 |
| Chemically etched | 0.32 | 750 hours dry $N_2$ | 0.6 |
| "B" Material | | | |
| Chemically cleaned | 0.49 | 1 hour air, dewpoint 14.5°C | 1.4 |
| Bead blasted | 0.15 | 1 hour air, dewpoint 12.2°C | 0.76 |
| Bead blasted | 0.76 | 1 hour dry $N_2$ | 2.35 |

## REFERENCES


1. R. W. Griessel, "Use of the mass spectrometer to evaluate the effectiveness of processing tube materials," ASTM Special Technical Publication No. 246, Symposium on Cleaning of Electronic Device Components and Materials (October 13-14, 1958).

2. J. Morrison, "Gas collection and analysis system employed in vacuum tube problems," Fourth National Symposium, Vacuum Symposium Transactions; pp. 100-103 (1957).

3. Peter F. Varardi, "Effect of pretreatment on the degassing of materials," Second International Congress on Vacuum Science and Technology, Transactions of the Eighth Vacuum Symposium; pp 73, 77 (1961).

4. R. W. Lawson, "Degassing properties of nickel," British Journal of Applied Physics 13, 115, 121 (1962).

5. L. Holland, "Problems in the construction and operation of bakeable and non-bakeable vacuum systems," Seventh National Symposium, Vacuum Symposium Transactions; p. 175 (1960).

6. B. D. Power and F. C. Robson, "Experiences with demountable ultra-high vacuum systems," Second International Congress on Vacuum Science and Technology, Transactions of the Eighth Vacuum Symposium; pp. 1175, 1176 (1961).

7. D. K. Das, "Outgassing characteristics of various materials in an ultra-high vacuum environment," Report No. AEDC-TDR-62-19, Arnold Engineering Development Center (January 1962).

8. F. A. von Flecken and H. G. Nöller, "Gasabgabe vershiedener metallischer hockvakuum-baustoffe," Second International Congress on Vacuum Science and Technology, Transactions of the Eighth Vacuum Symposium; pp. 58-65 (1961).

9. Chikura Hayashi, "Role of adsorption in production and measurement of high vacuum," Fourth National Symposium, Vacuum Symposium Transactions; pp. 13-26 (1957).

10. F. P. Bowden, F.R.S., and W. R. Throssel, "Adsorption of water vapor on solid surfaces," Proc. Royal Soc.; A 209, 279 (1951).