B. MECHANICAL DEVELOPMENT

The objectives of the mechanical development work have been to establish materials of construction for containing the melt that is a critical element of the process and to provide design bases for melt circulation, coal feeding and control of the process.

1. Corrosion Studies

Tests of the corrosion resistance of various materials were carried out by subjecting test pieces to molten sodium carbonate through which gases simulating the conditions to be used were passed through the salt which contained graphite, and gasification occurred during the test. In subsequent work, gases premixed to simulate process conditions were used. In all tests the molten salt contained coal ash and in most tests sodium sulfate or sodium sulfide was added to reflect the presence of sulfur during operation of the process. Gas velocities used were between 0.5 and 1.0 feet per second to ensure good mixing of the melt and to provide some velocity of the salt relative to the surface of the test piece. The diameter of the test units ranged from 2 to 4 inches.

The results of the corrosion tests are presented in Tables 5 and 6. The first of these tables presents the results obtained with Monofrax A which is a refractory of high purity fused alumina that is cast into molds in the molten state. The many hours of testing with very low corrosion rates ranging from 0.05 to 0.2 inches per year shown in Table 5 indicate that Monofrax A is an acceptable material for the intended services. One experiment (Test 10) gave a high rate of corrosion and the factors that could contribute to this result and which would have to be controlled in actual service were under investigation at the termination of the contract. As outlined in Section II-A-7, bench-scale experimentation on the solubility of alumina in sodium carbonate has indicated that very probably the carbon dioxide content of the aeration gas was insufficient to prevent the decomposition of sodium carbonate to sodium oxide. This latter compound most certainly would have attacked the Monofrax. Thus, decomposition should not be a problem in a commercial unit operating at about 400 psig since the partial pressure of carbon dioxide in both gasifier and combustor should be well above that required to prohibit sodium oxide formation. Thus, the main conclusion drawn from the corrosion work is that Monofrax A can withstand the reaction environment and is a satisfactory material of construction. In addition, preliminary vessel designs have been made in sufficient depth to ensure that Monofrax A linings of appropriate mechanical, as well as corrosion, characteristics can be built and operated. Also, preliminary tests on small blocks have demonstrated that an ultrasonic test technique is available to inspect Monofrax A blocks for flaws prior to their use in the vessels. Figures 21 through 24 show some typical lining details considered.

Thus, Monofrax A has demonstrated the ability to adequately withstand the test environments; it can be fabricated to the required shapes; and it can be checked for integrity. Accordingly, this material is considered to be satisfactory for the intended service.

Table 6 is a list of other materials which have been tested and found unsatisfactory because of unacceptably high corrosion rates, often high enough to destroy the samples completely.

TABLE 5

CORROSION TEST RESULTS

MONOFRAX A

TEST	DURATION	TYPE	TEMP.	AVERAGE CORROSION RATE INCHES PER YEAR	NOTE
3	200 hrs.	Gasification	1830-40°F	0.14	(1)
4	500 "	"	1830-40°F	0.18	(2)
7	395 "	"	1830-40°F	0.14	(3)
Oxy. #2	135 "	Simulated Combustion	1830-40°F	0.046	(4)
Oxy. #3	1000 "	"	1830-40°F	0.05	(5)
#9	407 "	Simulated Gasification	1840°F	0.31	(6)
#10	540 "	"	1840°F	1.52	(7)
#11	138 "	"	1840°F	0.0	(8)
#12A	72 "	"	1840°F	0.19	(9)

For notes please see following pages ....

NOTES FOR TABLE 5

(1) The three samples of Monofrax A came out of Test #3 relatively unaffected.

(2) Evaluation of Monofrax A samples from Test #4 indicates little corrosion. These samples seem to be as strong as before the test and show only a discoloration to differentiate the tested samples from the "as received".

(3) The Monofrax A withstood the test while other high purity aluminas did not. One possible explanation for this is in the manufacture of the Monofrax. Monofrax A is the only material that is melted and cast to shape; the other specimens are "all slip cast", i.e., cast then fused at high temperature.

(4) Monofrax A in very good condition at the end of the test.

(5) At the end of 85 hours the Monofrax A was in excellent condition, corrosion rate to this point was 0.01 in/yr. At the end of 200 hours the maximum total corrosion rate was 0.05 in/yr; after 500 hours, the average corrosion rate was found to be 0.03 in/yr and the sample was in good condition. After 846 hours, the sample was still in excellent condition, with an average corrosion rate of 0.08 in/yr. At the completion of the test, 1,000 hours, the overall average corrosion rate was found to be .05 in/yr. This is due to almost no corrosion from 846 to 1,000 hours, hence a lower average rate. The specimen, while still intact and in good condition, showed some areas of localized corrosion where the grain size was the largest.

continued . . .

(6) This was a simulated gasification test. The average corrosion rate was .31 in/yr. This rate is based on dimensions for the cracked specimens and is not really representative of the corrosion to be expected had the specimens remained whole. The cracking was most likely caused by the failure of the supports used to apply compression to adjacent test pieces. The metal rod passing through holes in the specimens to apply compressive stress expanded sufficiently on corrosion to crack the specimen

(7) These specimens were found to have corroded at an average rate of 1.52 in/yr, the reason for the high rate is felt to be connected with the composition of the feed gases.

(8) Test 11 was conducted using CO_2 only as the feed gas, as there will be in excess of 1 atmosphere of CO_2 in the actual gasifier. Corrosion rates of the Monofrax A were zero for this test.

(9) Test #12A was an exact duplicate of Run #10, to try to duplicate the corrosion rate. The samples when examined at 72 hours were found to have corroded at an average rate of 0.191 in/yr which is in keeping with results of runs prior to run #10. One possible explanation for the results of Run #10 is that the CO_2 concentration in the simulation gas mix was insufficient to inhibit the decomposition of Na_2CO_3 to Na_2O, which is quite capable of attacking Monofrax A. This factor is of course controllable by process conditions and at best is a marginal condition as demonstrated by the reasonable corrosion rates obtained in Run #12A. It is not considered a serious bar to the use of Monofrax A, expecially under the proposed operating conditions where the high CO_2 pressures should be sufficient to surpress Na_2O formation in contrast with the atmospheric test conditions.

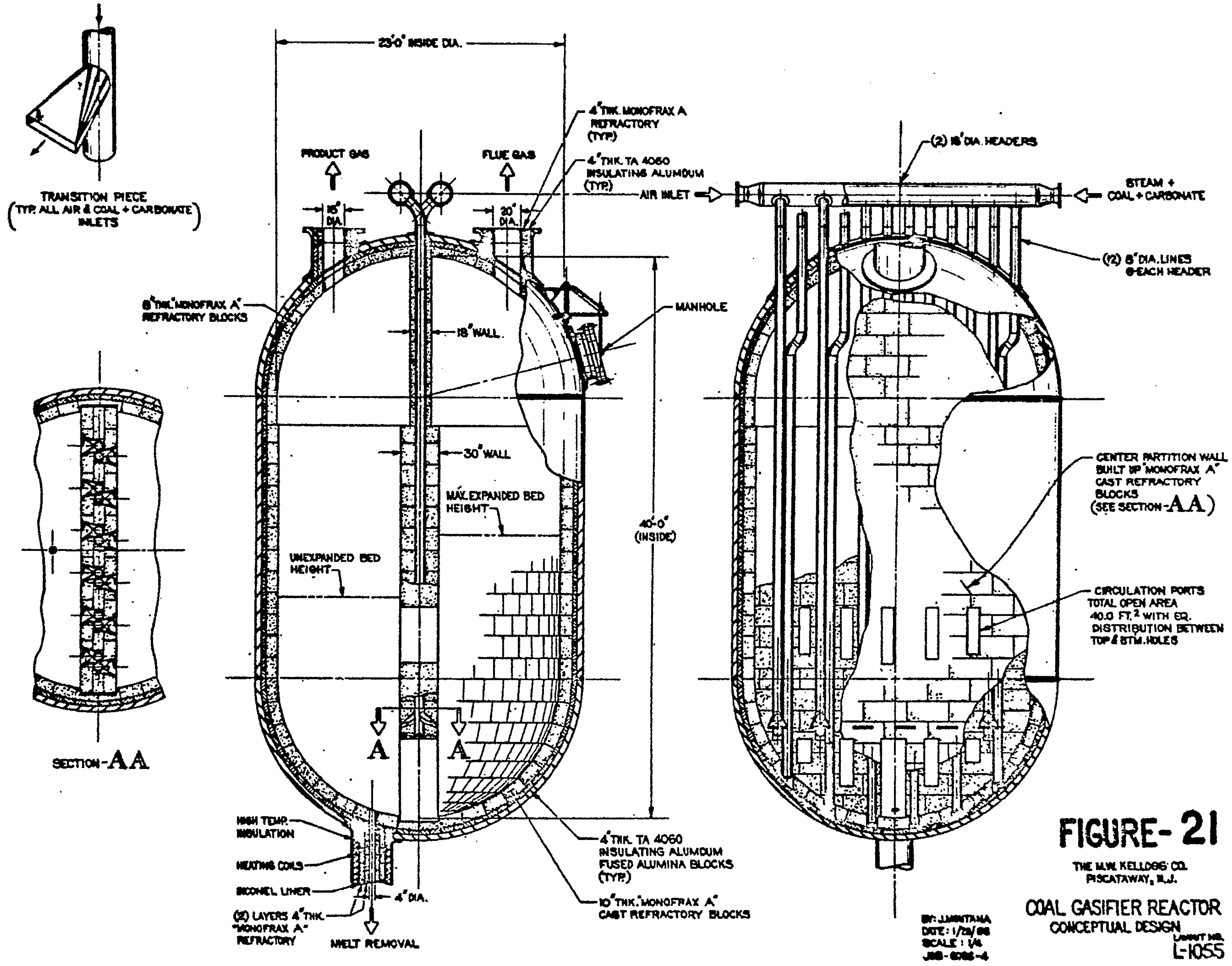
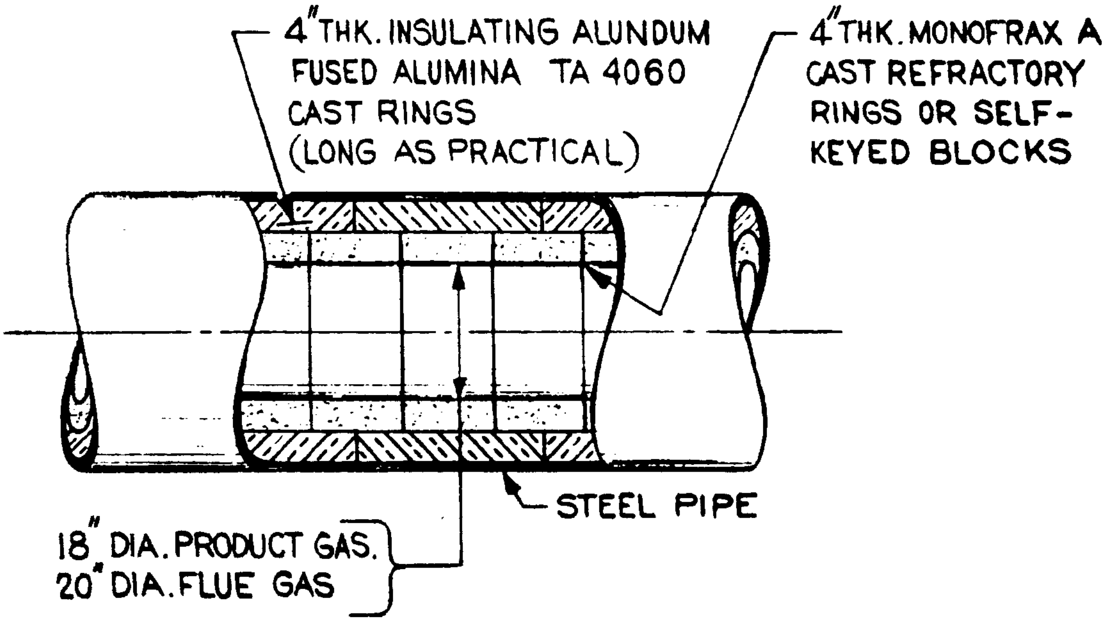


FIGURE- 21

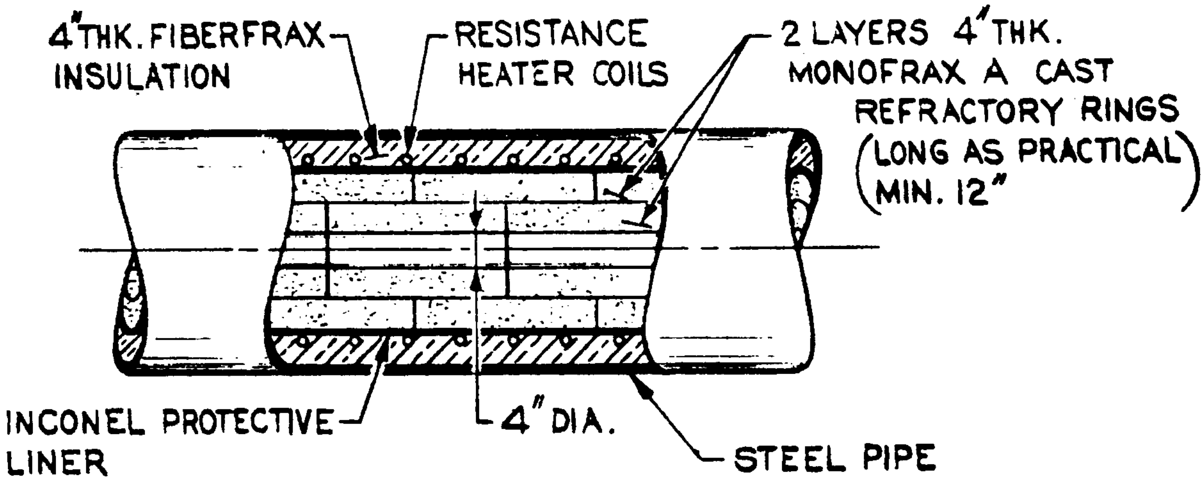
THE M.W. KELLOGG CO.
PISCATAWAY, N.J.

COAL GASIFIER REACTOR
CONCEPTUAL DESIGN

TRANSFER LINE CONSTRUCTION



PRODUCT GAS & FLUE GAS LINE (TYP.)



MELT TRANSFER LINE

FIGURE-22

J.M. 2/3/66

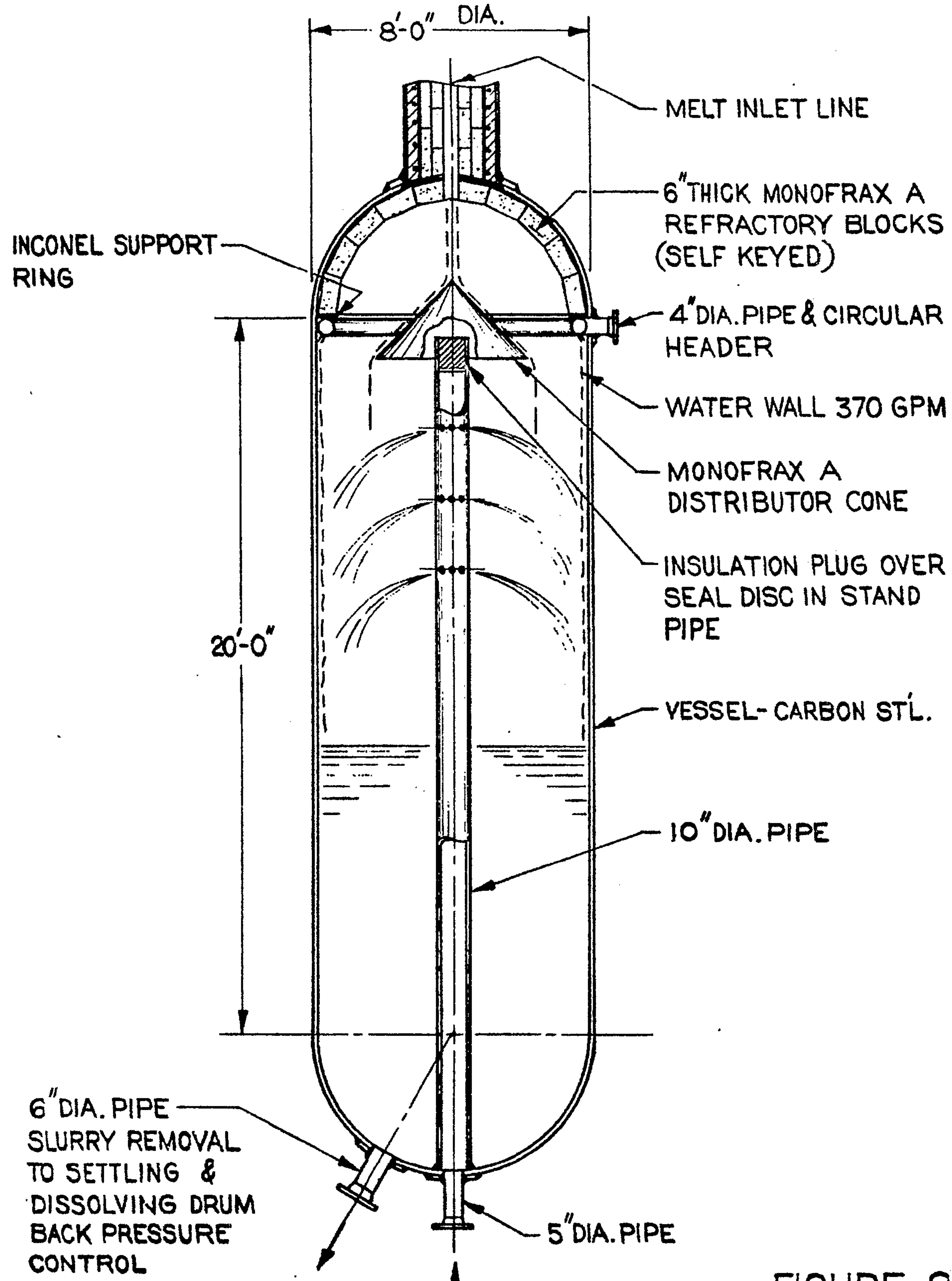


FIGURE-23

MELT QUENCH TOWER

J.M. 2/1/66

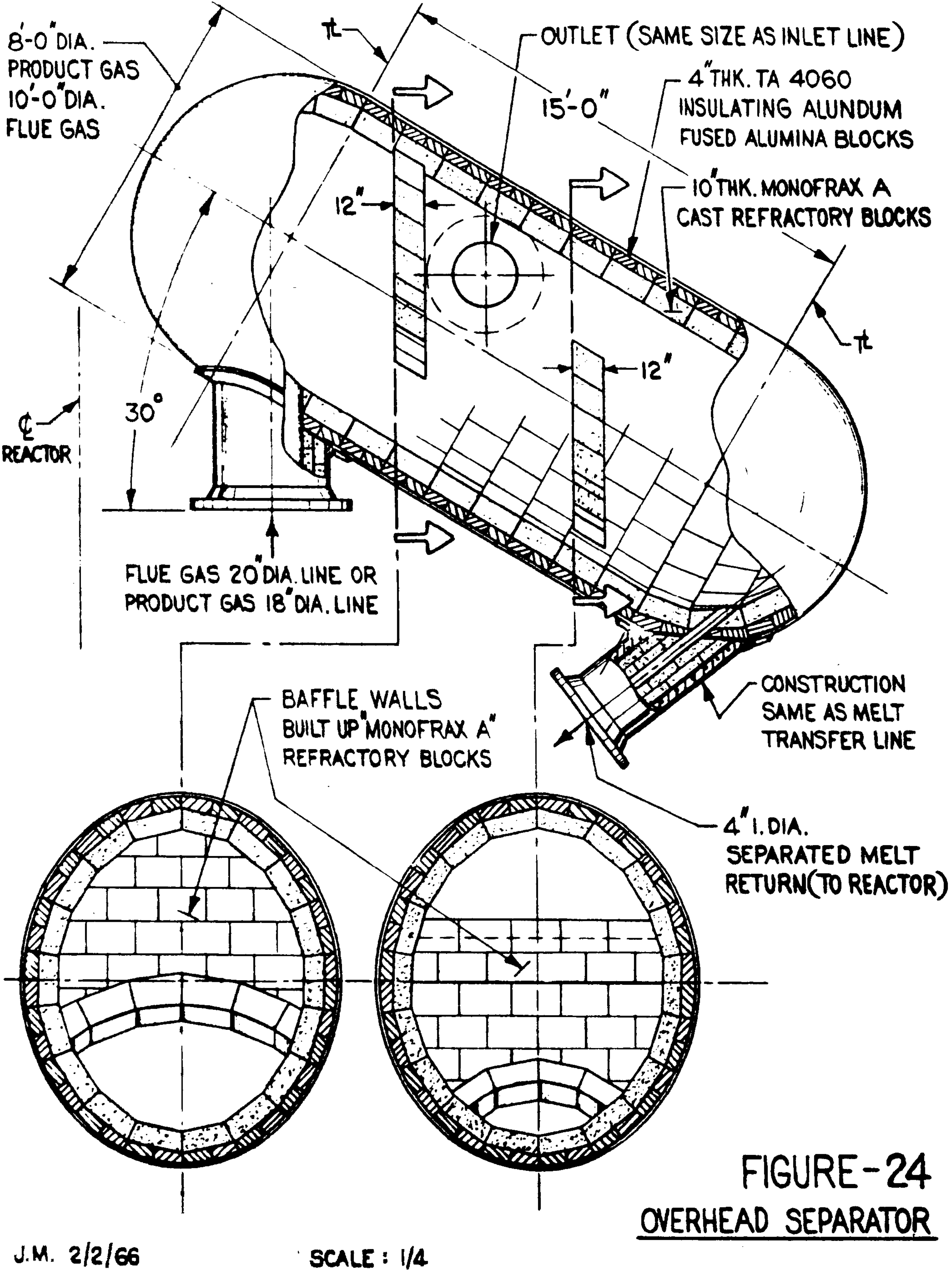
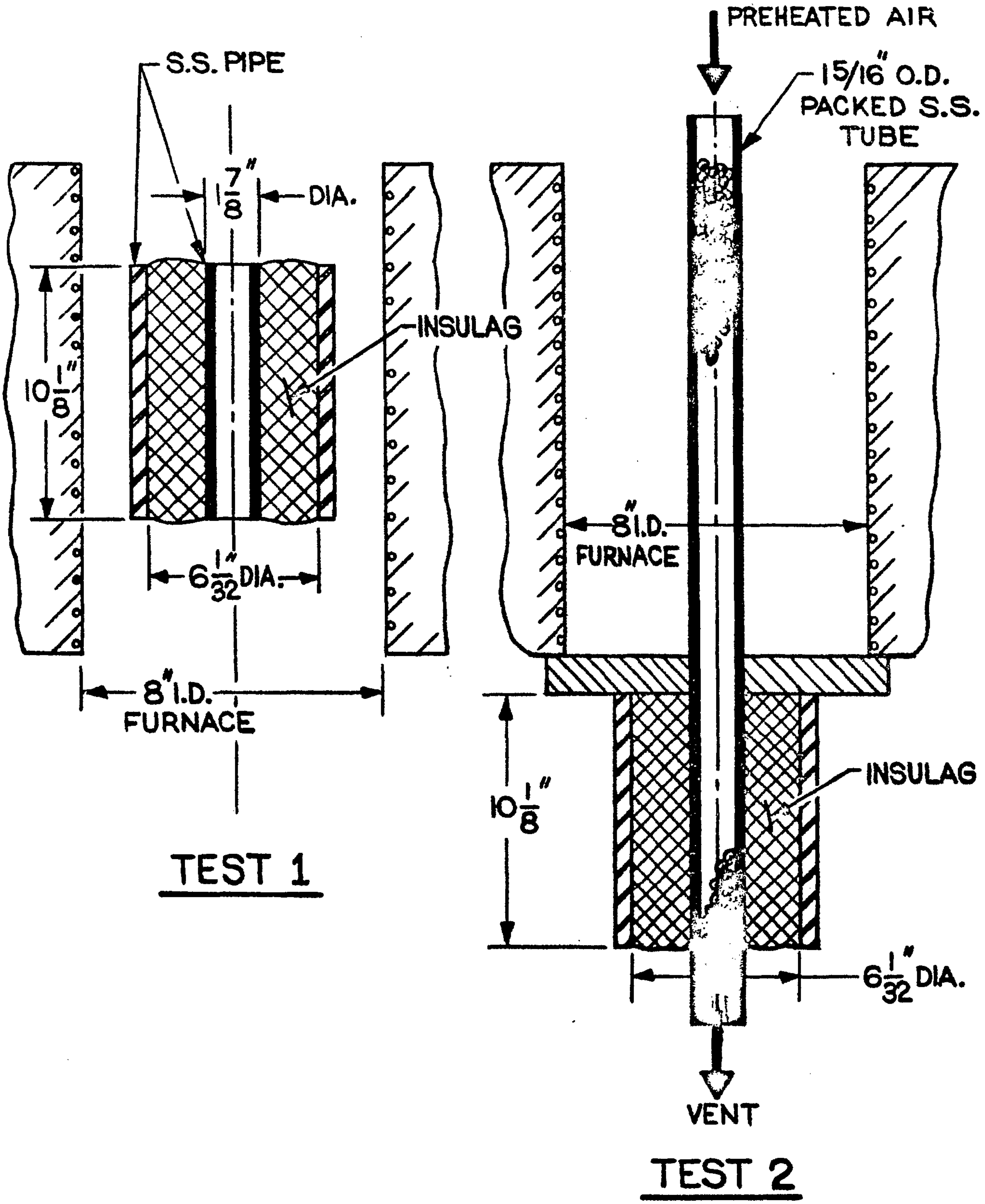


FIGURE - 24

OVERHEAD SEPARATOR

J.M. 2/2/66 SCALE : 1/4

FIGURE 25 INSULAG TEST



SCALE: NONE

TABLE 6

MATERIALS TESTED AND FOUND UNSATISFACTORY

METALS

MATERIAL DESIGNATION	MANUFACTURER
Thermalloy T-28	Electro-Alloys
Thermalloy T-38	Electro-Alloys
Thermalloy T-47	Electro-Alloys
Inconel 600	International Nickel Co.
Inconel 702	International Nickel Co.
Incoloy 800	International Nickel Co.
Incoloy 800 w/1.5% Al	International Nickel Co.
Incoloy 800 w/2.4% Al	International Nickel Co.
Incoloy 800 w/3.6% Al	International Nickel Co.
Incoloy 804	International Nickel Co.
Type 310 S. S.	International Nickel Co.
50% Cr. -50% Ni	International Nickel Co.
RA 333	Rolled Alloys
RA 446	Rolled Alloys
RA 600	Rolled Alloys
Hastelloy B	Union Carbide
Hastelloy C	Union Carbide
Haynes 150	Union Carbide
Molybdenum (Pure)	
Platinum (Pure)	
Tungsten (Pure)	

Table 6 continued . . . . .

Table 6 continued

CERAMICS

MATERIAL DESIGNATION	MANUFACTURER
Mullfrax H	Carborundum Company
Mullfrax W	Carborundum Company
Silicon Nitride	Carborundum Company
Zirofrax O	Carborundum Company
AD-99	Coors Electric Company
Lucalox	General Electric Company
Ritex C.B.	Grefco.
Monofrax K-3	Harbison-Carborundum
Monofrax M	Harbison-Carborundum
Chromex B	Harbison-Walker
Harbide	Harbison-Walker
Harklase	Harbison-Walker
Varnon B-F	Harbison-Walker
AP-35	McDanel
Triangle RR	Morganite
4995	Norton
5995	Norton
Zirconia 8-D	Zircoa
Zirconia 60-D	Zircoa
Zirconia RMF-5F	Zircoa
Zirconia Y-1027	Zircoa
Zirconia Y-1235	Zircoa
Zirconia Y-1484	Zircoa
Boron Nitride	Zircoa

MISCELLANEOUS

RA 600 w/Zirconia
Spray Coat

Two temperature effect tests were conducted on Insulag insulating material which had been proposed as a backup insulation behind Monofrax A in melt contact areas.

Test #1 subjected a hollow cylindrical section of Insulag encased in stainless steel pipe (except for the ends) to three temperature cycles of from 220 to 1850°F. Figure 25 shows the test section configuration utilized in Test 1 and Test 2. In Test 1, the entire assembly was heated in an electric furnace. Upon completion of the test, it was found that the Insulag had undergone severe shrinkage, checking and cracking. One longitudinal crack extended completely through the section of Insulag, thus significantly impairing its insulating capacity. The insulation itself had a hard crusty texture.

Test #2 subjected a hollow cylindrical section (similar to the test section in Test #1) to 13 cycles of from 220 to 1500°F on the inside wall. The outside wall in this case was exposed to the atmosphere and reached a maximum temperature of 275°F, thus subjecting the insulation to a radial temperature gradient similar to that which insulation would experience in service. In this case, the insulation remained in good condition except for a definite powdering of insulation extending into the insulation for 1/4" from from the hot surface. The outside appeared sound and, although not quite as hard as in Test #1, it was uncracked except for a closed circumferential crack which may have been produced mechanically on disassembly.

In summary, Insulag appears to be a satisfactory insulation at temperatures of 1500°F and below if the powdering can be tolerated.

2. Coal Feeding Studies

Proper measurement and control of coal feed rate is clearly important in the gasification process. Two schemes are proposed for this service; one, to use a calibrated star-wheel feeder to control the rate, and two, to use the pressure drop across a calibrated pipe section to measure the rate. These two systems would be used in conjunction with each other to provide a check on each.

Measuring the coal flow rate by pneumatic transport has been studied with the result shown in Figures 26 and 27. It was found that for a given coal size distribution, the pressure drop per foot of length does depend on the rate of coal being carried. It is clear that a range of operating conditions would be chosen that would provide sufficient sensitivity of measurement for good control. Similar studies were conducted on sand.

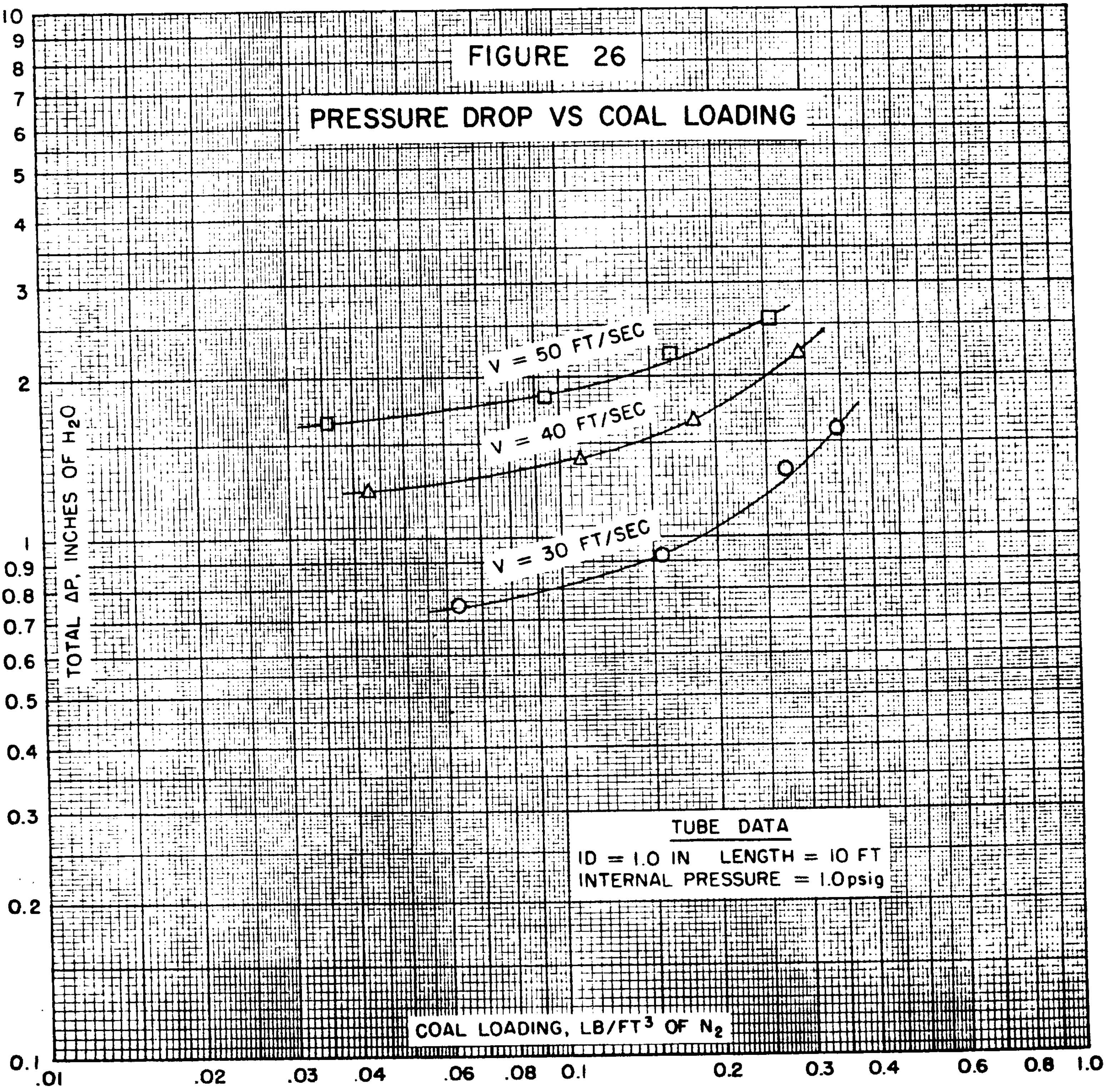


FIGURE 26
PRESSURE DROP VS COAL LOADING
V = 50 FT/SEC
V = 40 FT/SEC
V = 30 FT/SEC
TUBE DATA
ID = 1.0 IN LENGTH = 10 FT
INTERNAL PRESSURE = 1.0 psig
TOTAL ΔP, INCHES OF H₂O
COAL LOADING, LB/FT³ OF N₂
10
9
8
7
6
5
4
3
2
1
0.9
0.8
0.7
0.6
0.5
0.4
0.3
0.2
0.1
.01
.02
.03
.04
.06
.08
0.1
0.2
0.3
0.4
0.6
0.8
1.0

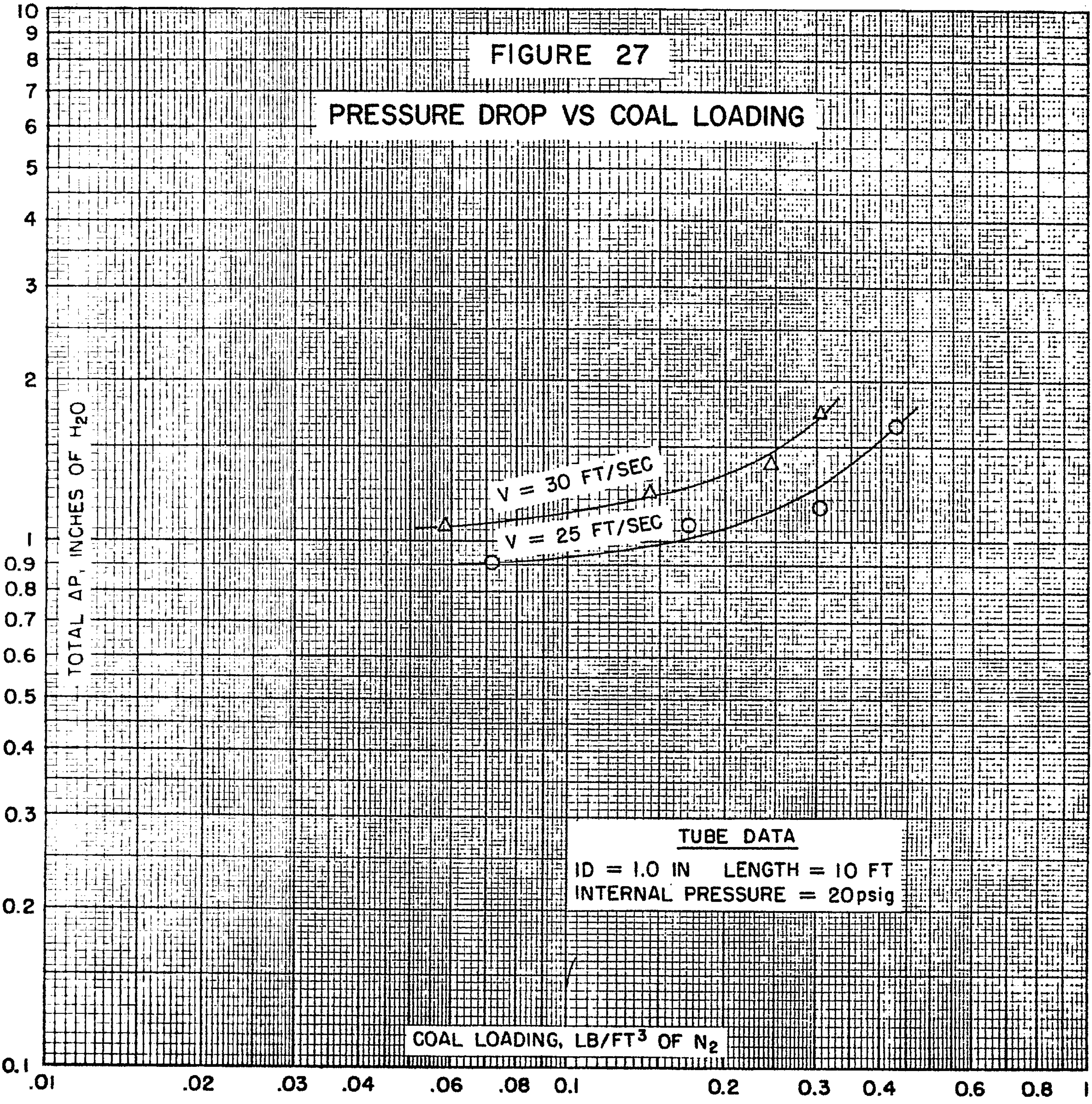


FIGURE 27

PRESSURE DROP VS COAL LOADING

TUBE DATA

ID = 1.0 IN LENGTH = 10 FT
INTERNAL PRESSURE = 20psig

Figure 28 shows the design details of a laboratory star-wheel feeder developed for coal feeding of particles up to 10 mesh. Figure 29 is a calibration curve of this feeder for various size cuts of coal at 0 and 400 psig pressure.

3. Quench Tests

Melt, containing coal ash, has been discharged through orifices up to 3/4" in diameter into cold stagnant water of varying depths. Some large agglomerations were observed when the water depth was less than 4 inches. Depths greater than 4 inches of water caused the melt to form high surface-to-volume ratio particles which resembled popcorn. This "popcorn" rapidly decomposed if left in the water until a thin slurry remained. Depths of water over 9" seemed to have little further effect on the quenched material.

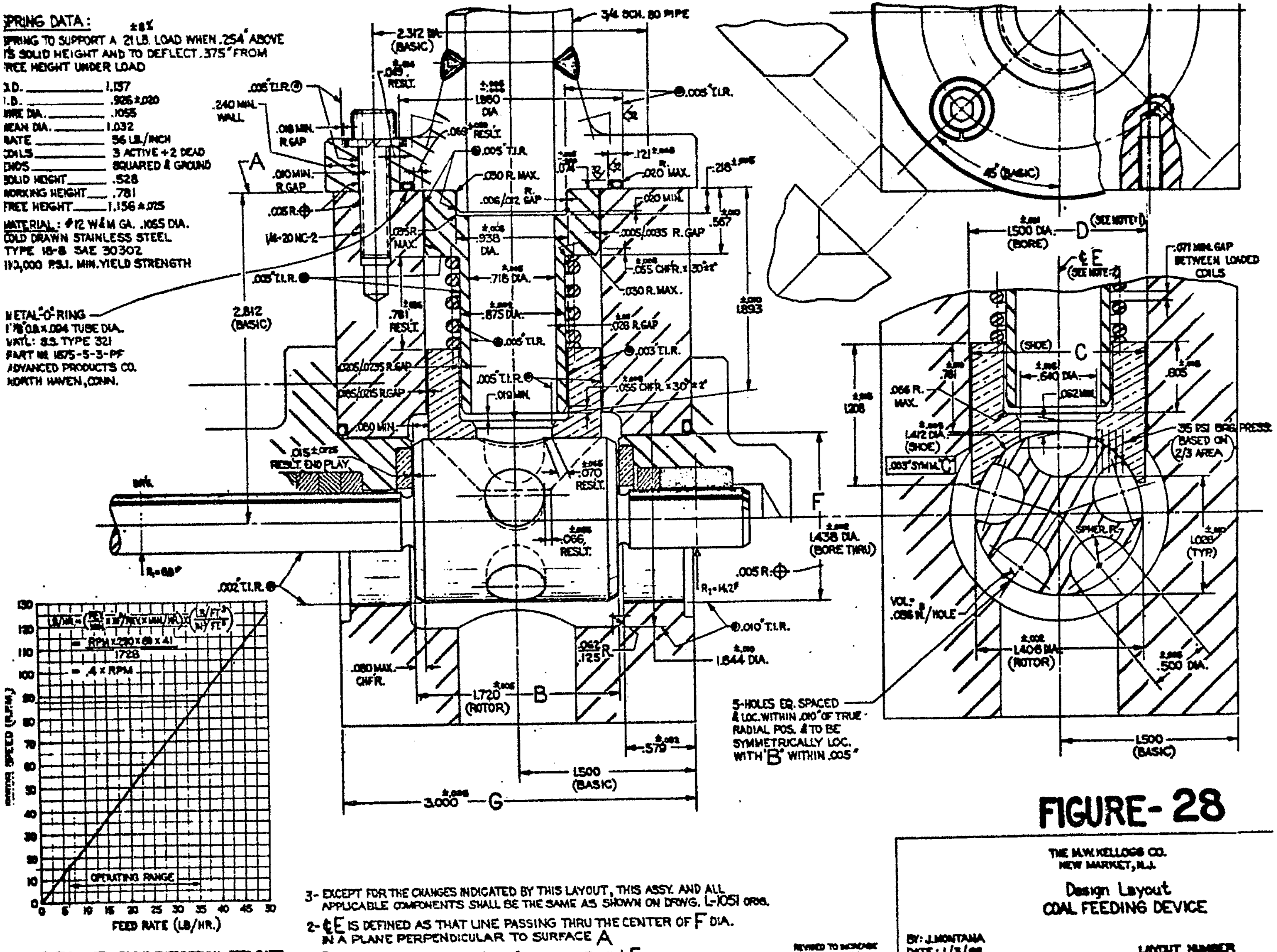
If the quenched melt is allowed to stand in water at temperature for some period of time, grinding of the slurry may not be required.

4. Cross-Flow Contamination

Circulation of molten salt between the gasifier and combustion vessel may result in cross-flow contamination of exit gas streams by entrained or dissolved gas carried by the melt. In an effort to determine the order of magnitude of cross flow contamination that might be expected, tests were carried out in a cross-flow circulation model. Figure 30 shows the test apparatus used. Oxygen was used for gas lift and aeration of the liquid in the vessel on one side and nitrogen for the other. Contamination was measured as a percent of nitrogen appearing in the outlet gas on the oxygen side by means of gas chromatography.

Tests were conducted with aeration superficial velocities from 0 to 2.0 feet per second with liquid transfer line velocities from 0.77 to 2.23 feet per second using water and ethylene glycol as simulated melt materials. The results of these tests are shown on Figures 31, 32, and 33.

The results of these tests were quite encouraging. The cross-flow contamination levels experienced in the model were within the order of magnitude of contamination levels envisioned as acceptable for the actual process. Actual cross-flow contamination will be dependent on circulation rate requirements, solubility of contaminants in the melt and physical design of the vessel.



SPRING DATA: ±8%
SPRING TO SUPPORT A 21 LB. LOAD WHEN .254" ABOVE
IT'S SOLID HEIGHT AND TO DEFLECT .375" FROM
FREE HEIGHT UNDER LOAD

O.D. ____________ 1.157
I.D. ____________ .926 ±.020
WIRE DIA. ________ .1055
MEAN DIA. ________ 1.032
RATE ____________ 56 LB./INCH
COILS ___________ 3 ACTIVE + 2 DEAD
ENDS ____________ SQUARED & GROUND
SOLID HEIGHT _____ .528
WORKING HEIGHT ___ .781
FREE HEIGHT ______ 1.156 ±.025

MATERIAL: #12 W&M GA. .1065 DIA.
COLD DRAWN STAINLESS STEEL
TYPE 18-8 SAE 30302
112,000 P.S.I. MIN. YIELD STRENGTH

METAL "O" RING
1 7/8" O.D. x .094 TUBE DIA.
MATL: S.S. TYPE 321
PART NO. 1875-5-3-PF
ADVANCED PRODUCTS CO.
NORTH HAVEN, CONN.

ROTOR SPEED VERSUS THEORETICAL FEED RATE
(BASED ON 41 LB/FT³ COAL DENSITY
& ROTOR CAPACITY OF .280 IN³/REV.)

3- EXCEPT FOR THE CHANGES INDICATED BY THIS LAYOUT, THIS ASSY. AND ALL APPLICABLE COMPONENTS SHALL BE THE SAME AS SHOWN ON DRWG. L-1051 ORIG.

2- ¢ E IS DEFINED AS THAT LINE PASSING THRU THE CENTER OF F DIA. IN A PLANE PERPENDICULAR TO SURFACE A

1- D DIA. TO BE LOCATED WITHIN .003" EITHER SIDE OF ¢ E AND TO BE SYMMETRICAL WITH G WITHIN .003"

NOTES:

FIGURE- 28

THE M.W. KELLOGG CO.
NEW MARKET, N.J.

Design Layout
COAL FEEDING DEVICE

REVISED TO INCREASE
FEED CAPACITY
REV. "A" DESIGN FEED
RATE WAS 15 LB/HR
@ 36 RPM

BY: J. MONTANA
DATE: 1/3/[illegible]
SCALE: 2/1
JOB 6026-4

LAYOUT NUMBER
L-1051 REV. B

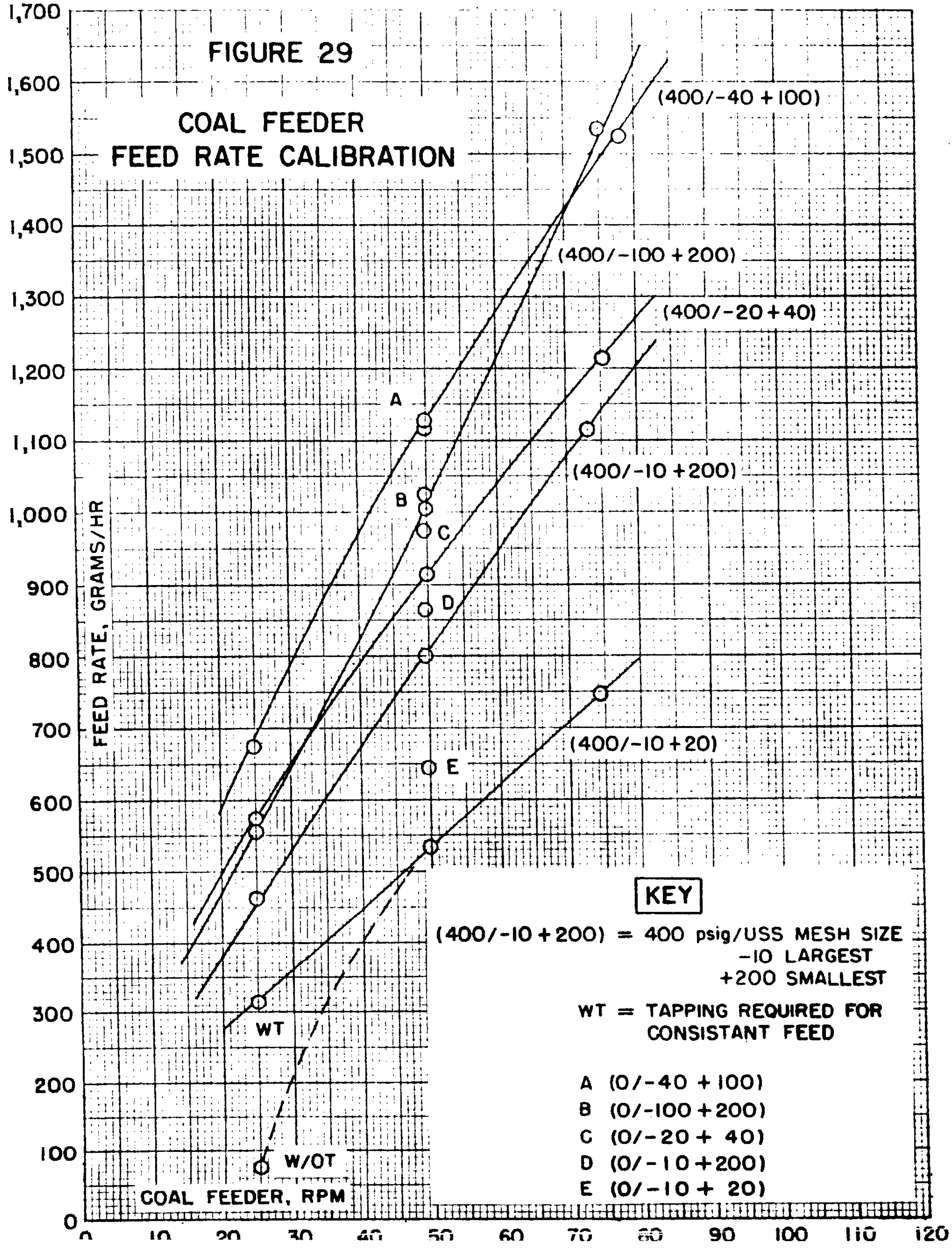
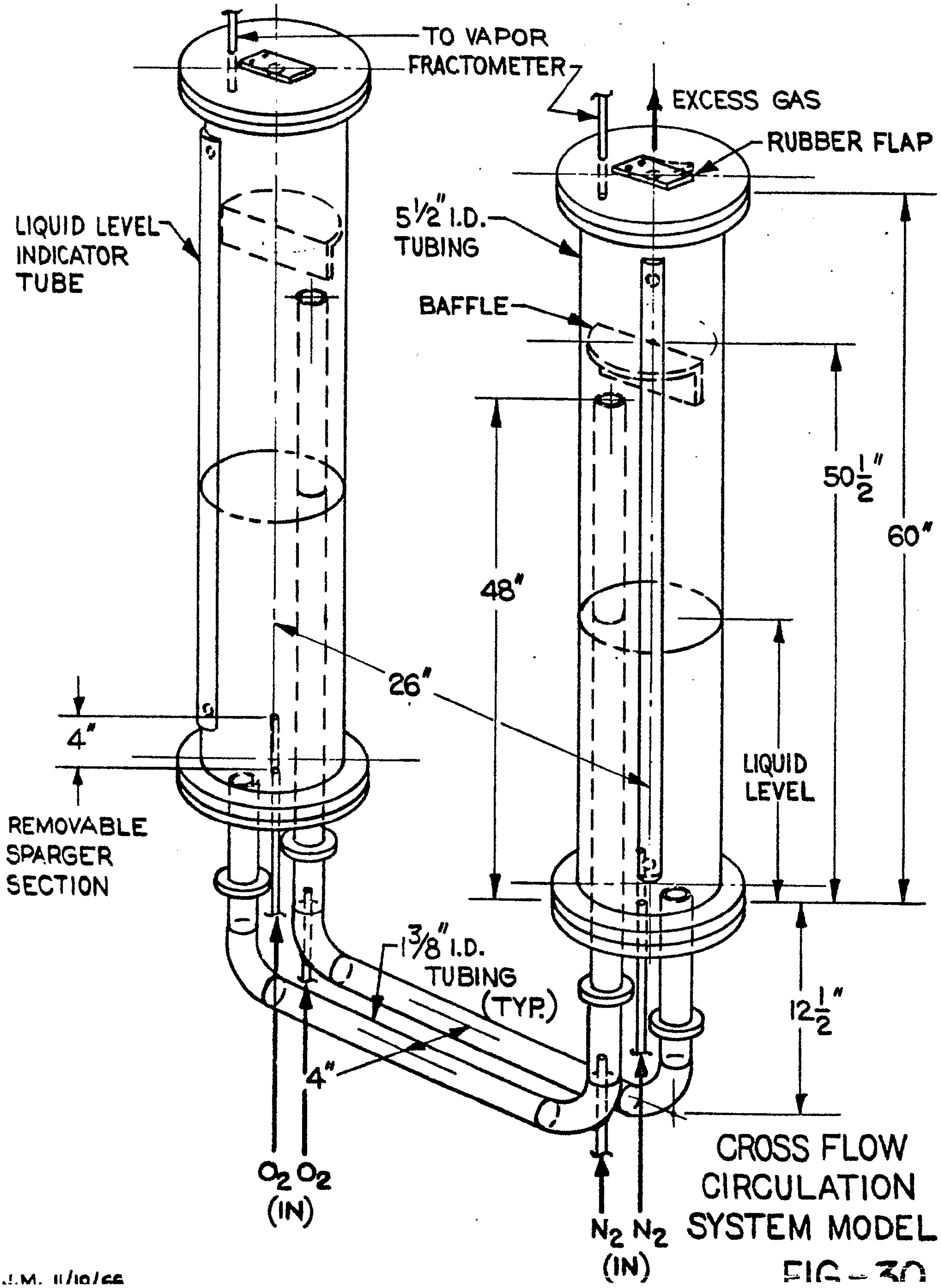


FIGURE 29

COAL FEEDER
FEED RATE CALIBRATION



CROSS FLOW CIRCULATION SYSTEM MODEL

FIG-30

J.M. 11/10/66

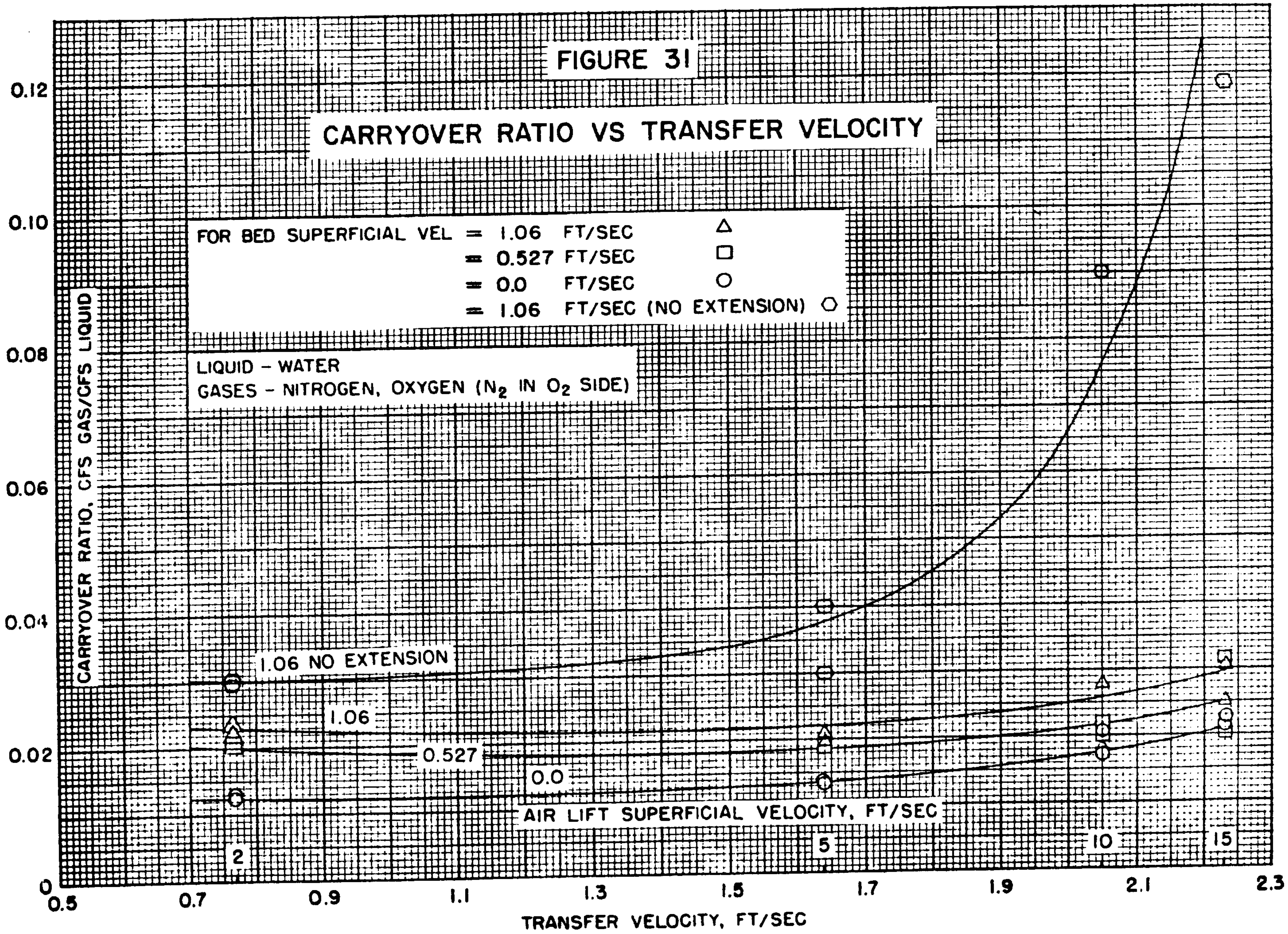


FIGURE 31

CARRYOVER RATIO VS TRANSFER VELOCITY

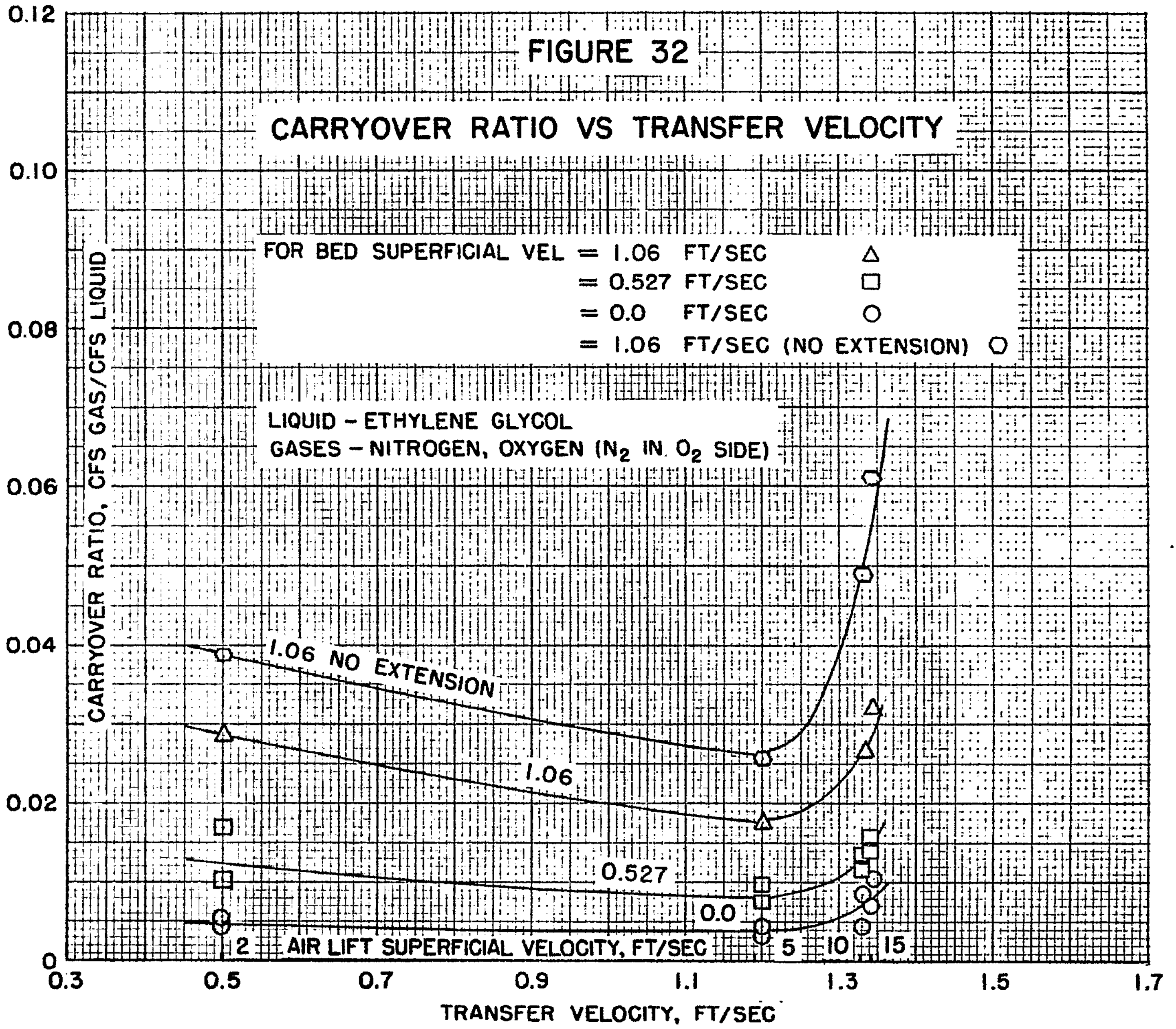


FIGURE 32

CARRYOVER RATIO VS TRANSFER VELOCITY

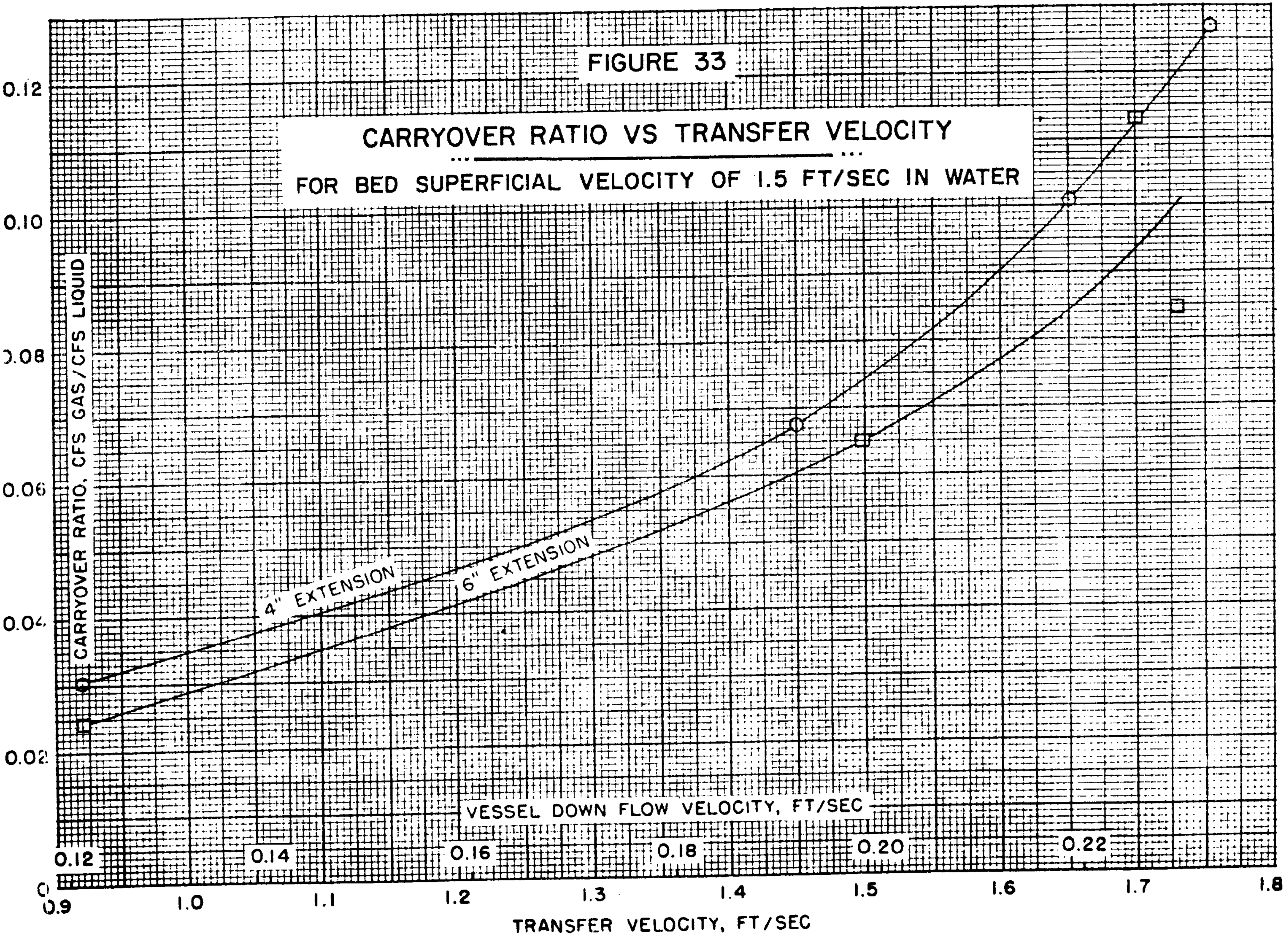


FIGURE 33

CARRYOVER RATIO VS TRANSFER VELOCITY

FOR BED SUPERFICIAL VELOCITY OF 1.5 FT/SEC IN WATER

5. Salt Transport

Studies have shown the "gas lift" to be the most suitable method of transporting the molten salt from the oxidation to the gasification side of the process and vice versa. A reasonable method, utilizing the Martinelli-Lockhart pressure drop correlation for two-phase, two-component flow in pipe, has been developed to predict the circulation and to size transfer lines.

Experimental verification of the method in the test apparatus shown in Figure 30 and in similar apparati has shown that the transfer rates can be predicted within ± 25 percent for fluids with viscosities in the range of 1 to 25 centipoise. Experimental results indicate the predicted diameter of the transfer line will be conservative for higher viscosity fluids (above 25 centipoise) in small diameter lines. However, based on observation of the gas lift bubble patterns, it is anticipated that results similar to the lower viscosity fluids can be obtained with line sizes of 4 inches and over. Figure 34 shows typical experimental vs. predicted pump rates for water.

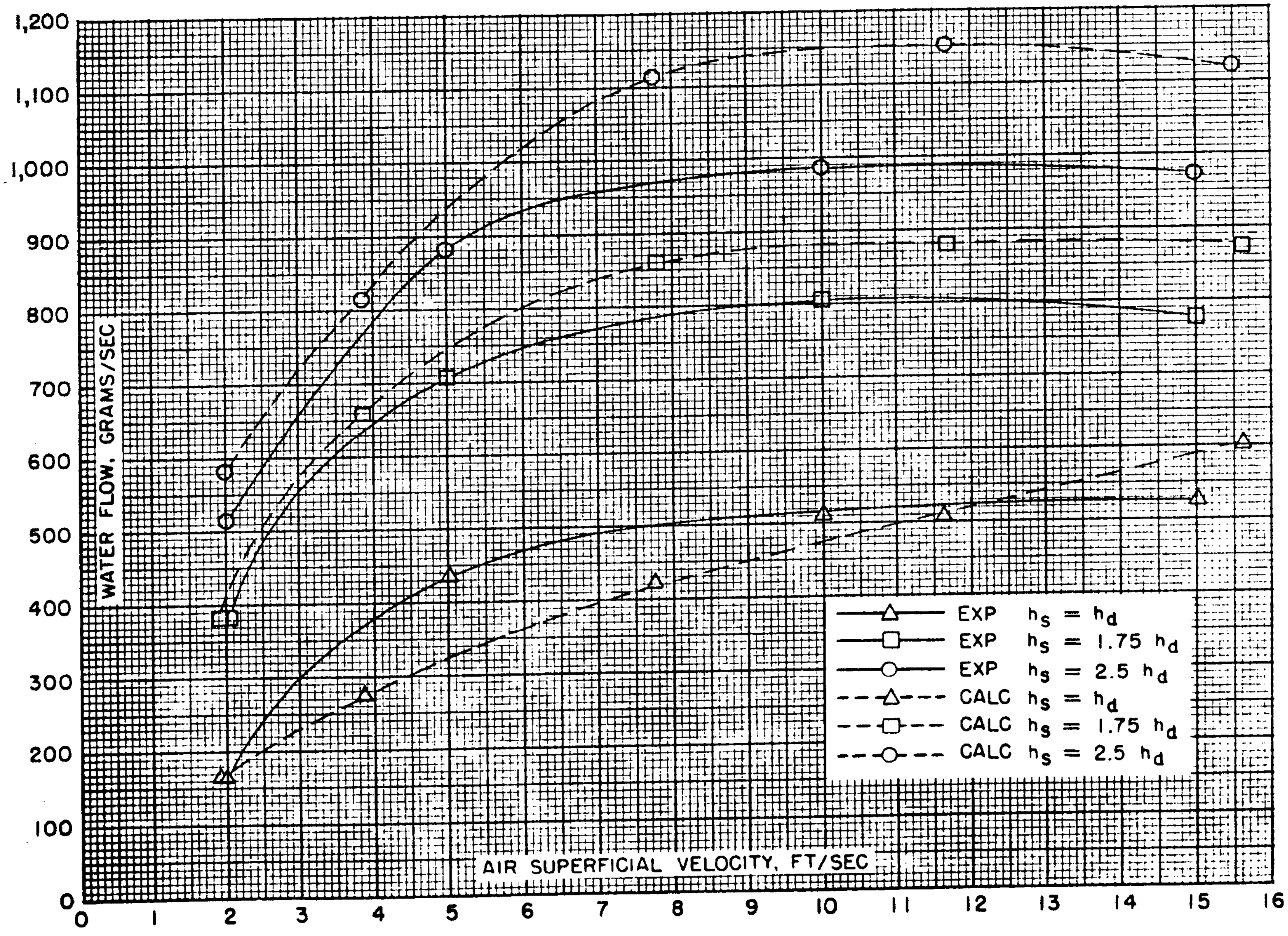
The calculation procedure has been programmed and used to size the pilot plant transfer lines and predict performance of the gas lift. Figures 35 and 36 show the performance curves developed for the currently proposed pilot plant configuration for both steam and air lift. From this data the transfer lines have been sized at 8 inches and the transport steam quantities needed here found to be a small portion of the total gasification steam requirements. Figure 37 shows the basic configuration and nomenclature used in the gas lift circulation program. Figure 38 shows the experimental flow vs. the program predicted flow for water and 100 cp. glycerine-water mix in the circulation model shown on Figure 30.

The program can handle additional bends in the horizontal transfer line to allow layout flexibility. Since the program handles each portion of the system individually and reports pressure drops for each, many other configurations are possible by the use of simple artifices.

FIGURE 34

EXPERIMENTAL VS CALCULATED FLOW

AIR LIFT PUMPING WATER 1-3/8" I.D. X 9' LONG TUBE



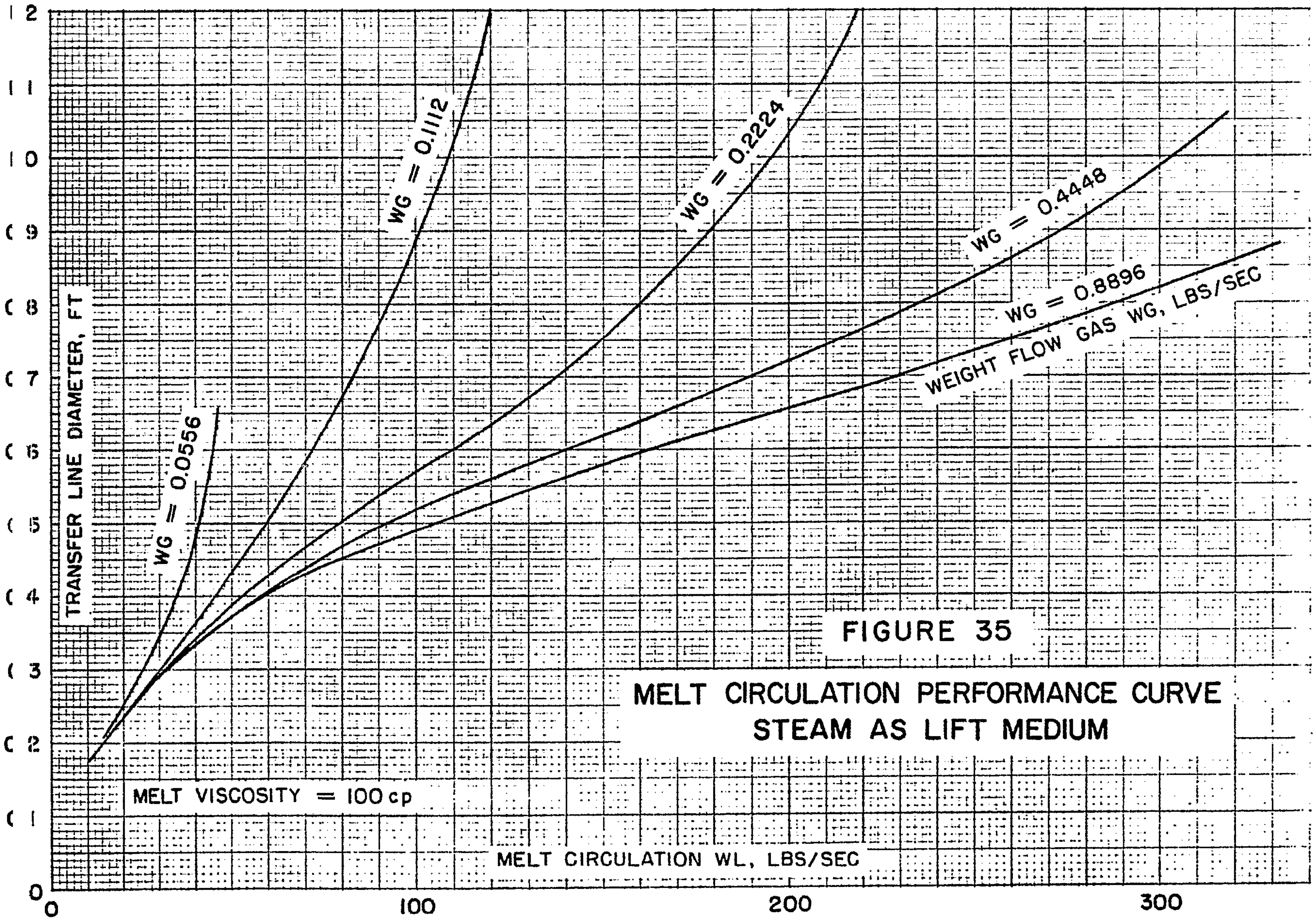


FIGURE 35

MELT CIRCULATION PERFORMANCE CURVE
STEAM AS LIFT MEDIUM

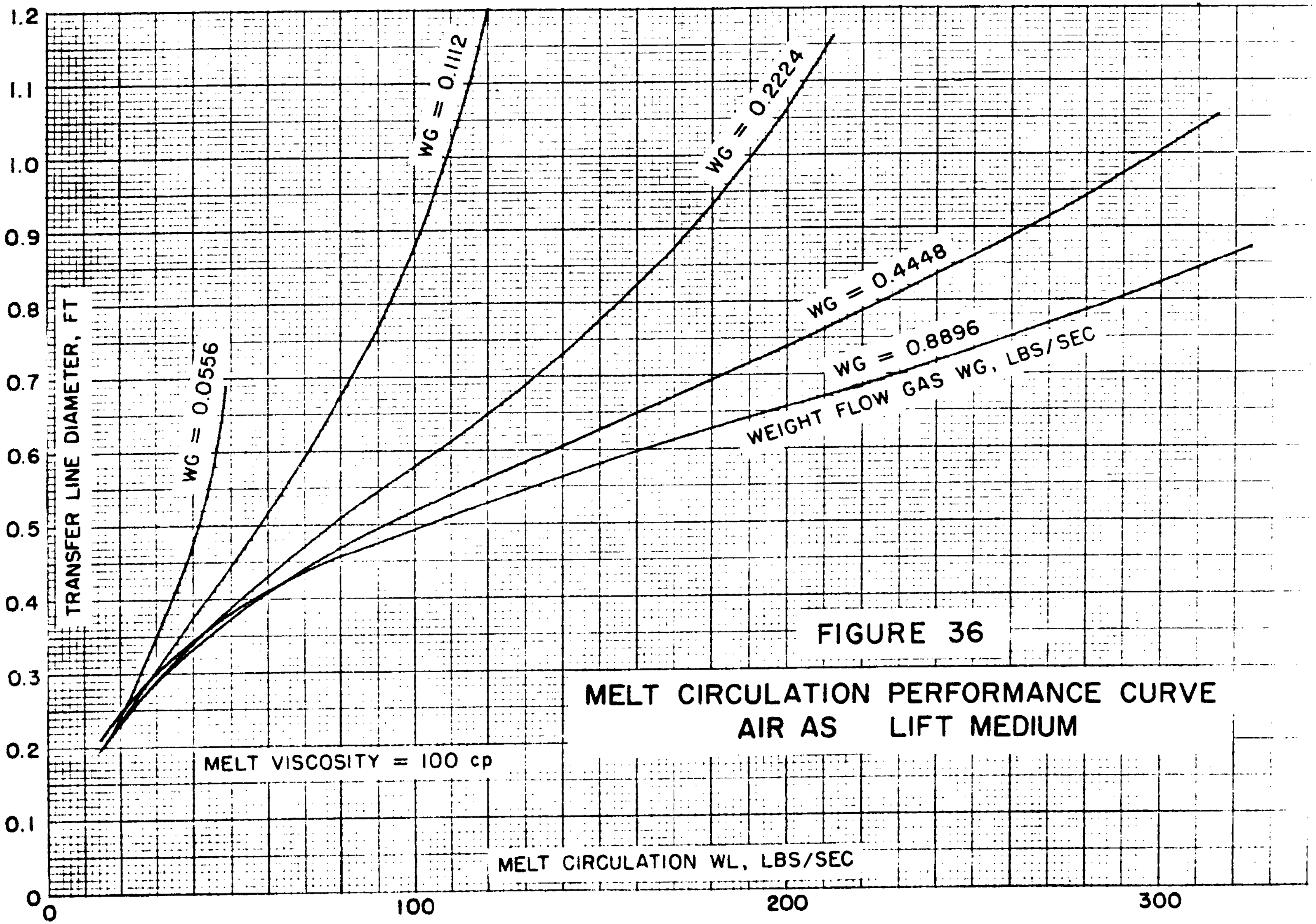
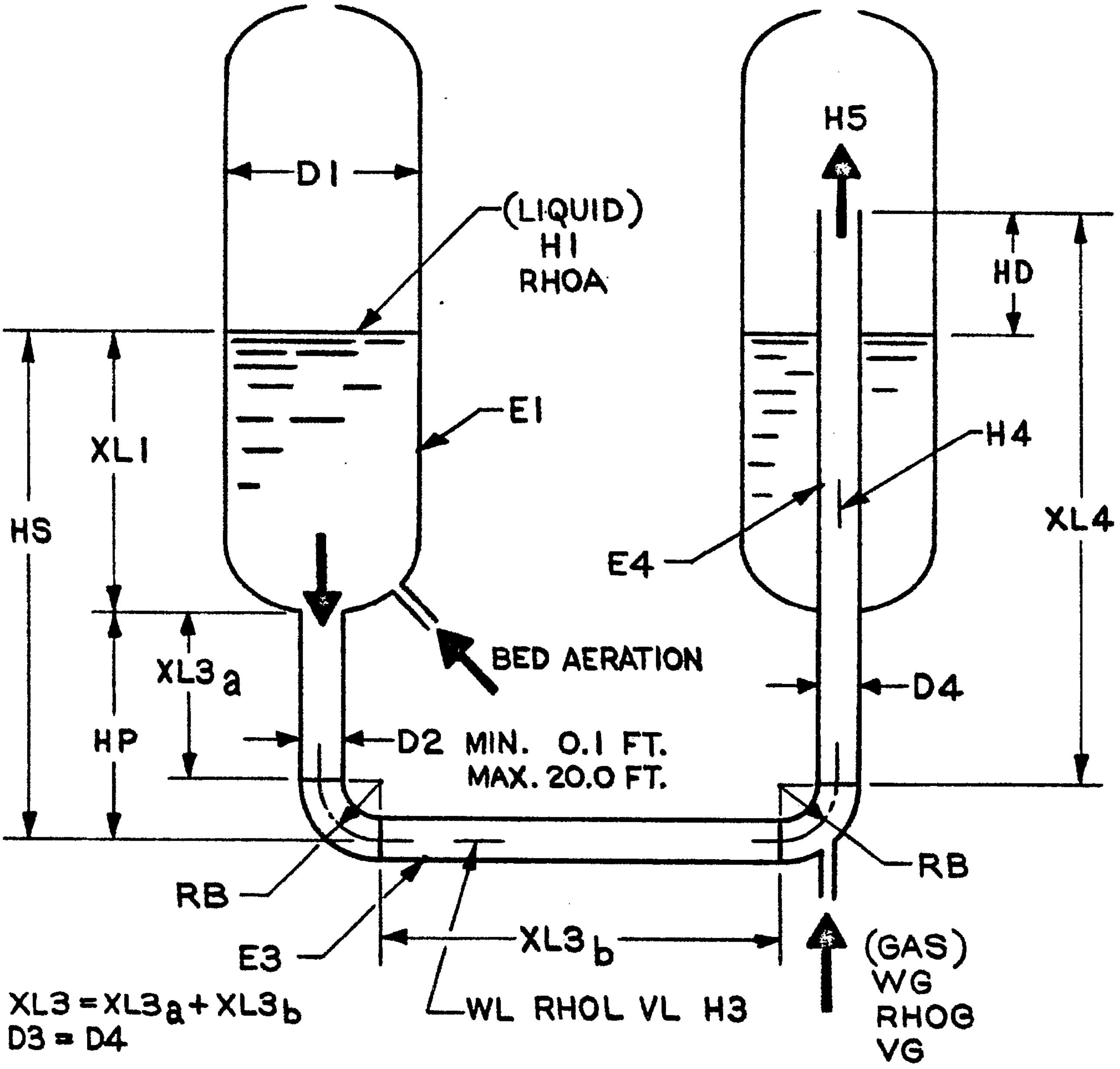


FIGURE 36

MELT CIRCULATION PERFORMANCE CURVE
AIR AS LIFT MEDIUM

FIGURE 37 GAS LIFT CIRCULATION SYSTEM



WL = Wt. OF LIQUID FLOWING (LBS./SEC.)

WG = Wt. OF GAS FLOWING (LBS./SEC.)

RHOG, RHOL, RHOA = DENSITY OF GAS, FLOWING LIQUID, VESSEL LIQUID (LBS./CU. FT.)

VG, VL = VISCOSITY OF GAS, LIQUID (LBS./FT./SEC.)

E1, E3, E4 = INSIDE SURFACE ROUGHNESS (Abs. FT.)

H1, H2, H3, H4, H5 = PRESSURE DROPS (FT. LIQUID)

HD, HP, HS, XL1, XL3 a, XL3 b, XL4 = HEIGHTS (FT.)

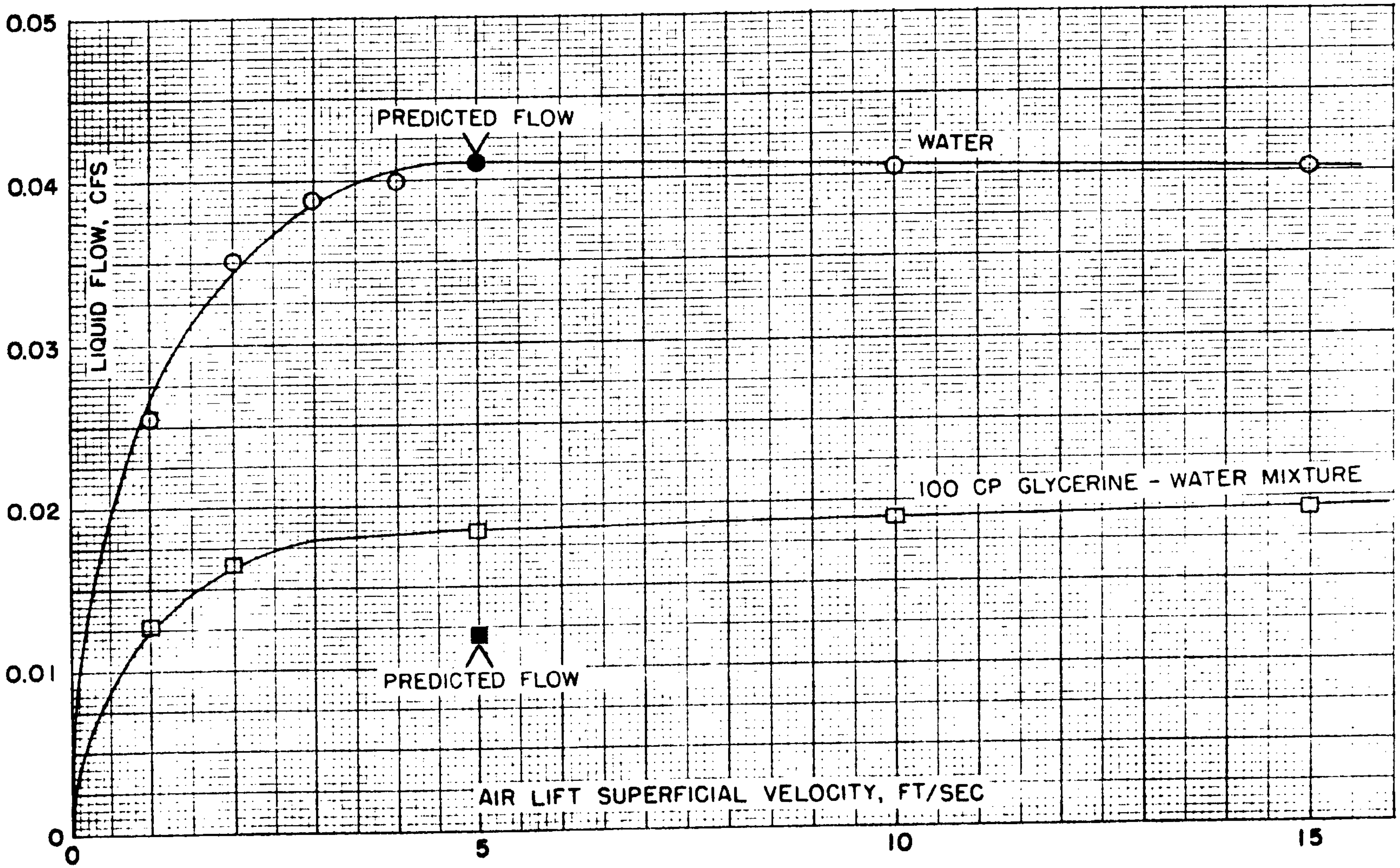
D1, D2, D3, D4 = INSIDE DIAMETERS (FT.)

RB = CENTER LINE BEND RADIUS (FT.)

FIGURE 38

EXPERIMENTAL FLOW RESULTS FOR 0 LIFT

CROSS FLOW MODEL — WATER AND 100 CP GLYCERINE MIXTURE



6. Bed Expansion Tests

a. Melt Studies

A series of tests was conducted in a 4-1/4-inch I. D. reactor to determine bed expansion of molten sodium carbonate with various superficial velocities of steam, nitrogen, and carbon dioxide. The effect of carbon level and ash concentration on bed expansion was also explored. Expansion of the bed with continuous coal feed with 7% ash and 4% carbon level in the bed was investigated.

All the above experiments were conducted with the reactor top removed to allow observation of the bed and permit measurement of bed expansion by use of a dip stick.

Figure 39 shows the effect of superficial velocity on bed height of molten sodium carbonate using steam, nitrogen or carbon dioxide as the fluidizing gas. Figure 40 displays the same information as a percent expansion.

It is interesting to note from these curves that nitrogen appears to give a 10 to 20 percent greater bed expansion for a given superficial velocity than either CO_2 or steam. Due to the obvious inaccuracies of measuring a bubbling bed height by a dip stick method, the data should be taken to indicate only a probable effect of fluidizing gas on bed expansion for a given superficial velocity. All plot points for the above curves are an average of three or more individual readings. Variations in individual readings averaged ± 10%; however, in extreme cases at the higher superficial velocities, variations of ± 25% were noted.

Figure 41 shows the effect with time of additions of 1, 2, 3, and 4 weight percent of coke in a molten sodium carbonate bed being agitated by nitrogen at 0.1 feet per second superficial velocity. The data indicate a rapid increase in bed expansion possibly resulting from liberation of volatile matter in the coke. After approximately four minutes, the bed level returned to the level produced by the agitation gas. A higher superficial velocity of 0.5 fps was introduced after 10 to 15 minutes of each run to establish its effect with carbon level. No significant variation was noted with carbon content. It therefore appears that, within the range of up to 4 percent carbon, carbon has little effect on bed expansion.

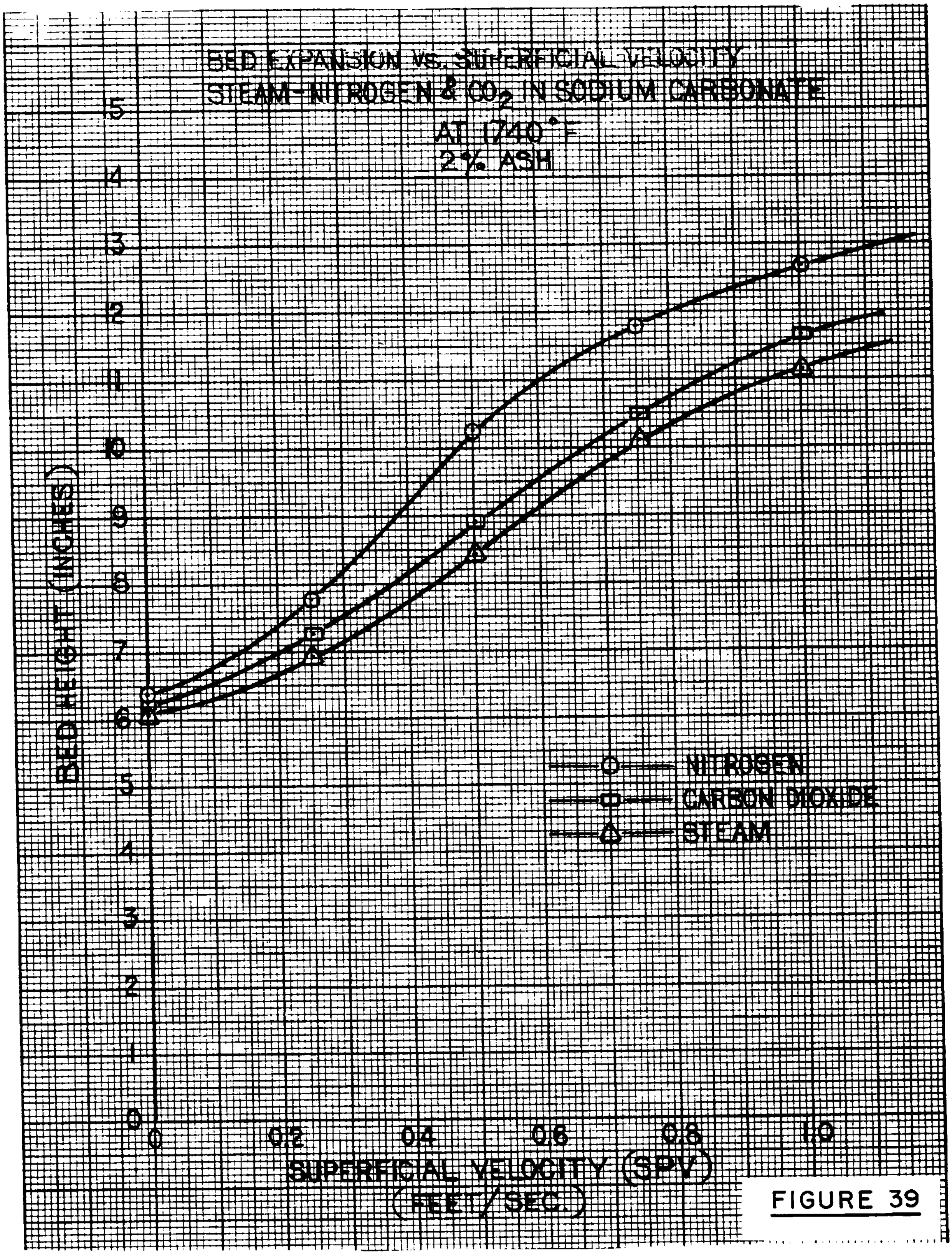

BED EXPANSION vs. SUPERFICIAL VELOCITY
STEAM-NITROGEN & CO_2 IN SODIUM CARBONATE
AT 1740°F
2% ASH
BED HEIGHT (INCHES)
0
1
2
3
4
5
6
7
8
9
10
11
12
13
14
15
0
0.2
0.4
0.6
0.8
1.0
SUPERFICIAL VELOCITY (SPV)
(FEET/SEC.)
NITROGEN
CARBON DIOXIDE
STEAM

FIGURE 39

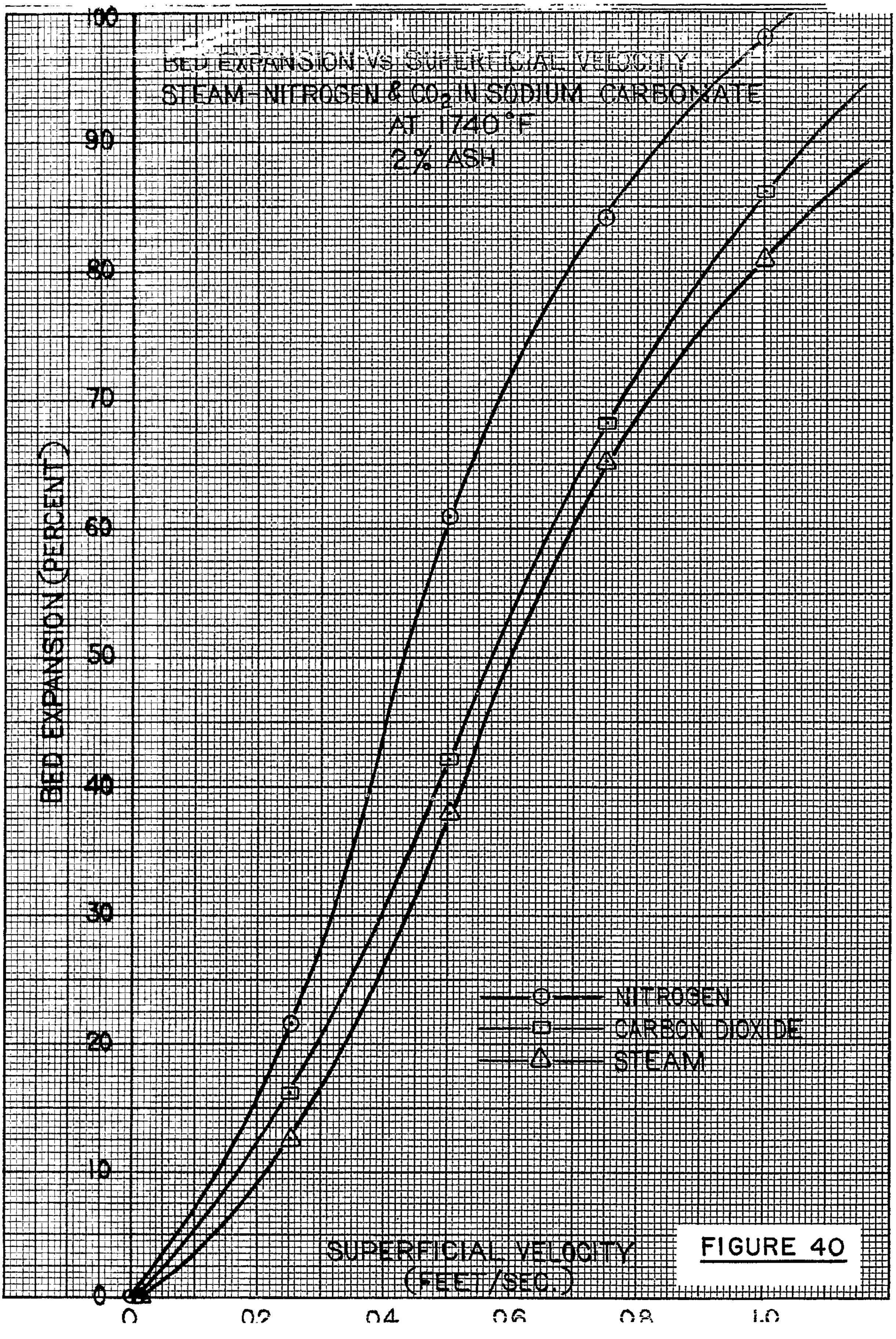

BED EXPANSION VS SUPERFICIAL VELOCITY
STEAM-NITROGEN & CO_2 IN SODIUM CARBONATE
AT 1740°F
2% ASH
BED EXPANSION (PERCENT)
100
90
80
70
60
50
40
30
20
10
0
SUPERFICIAL VELOCITY
(FEET/SEC.)
0
0.2
0.4
0.6
0.8
1.0
NITROGEN
CARBON DIOXIDE
STEAM

FIGURE 40

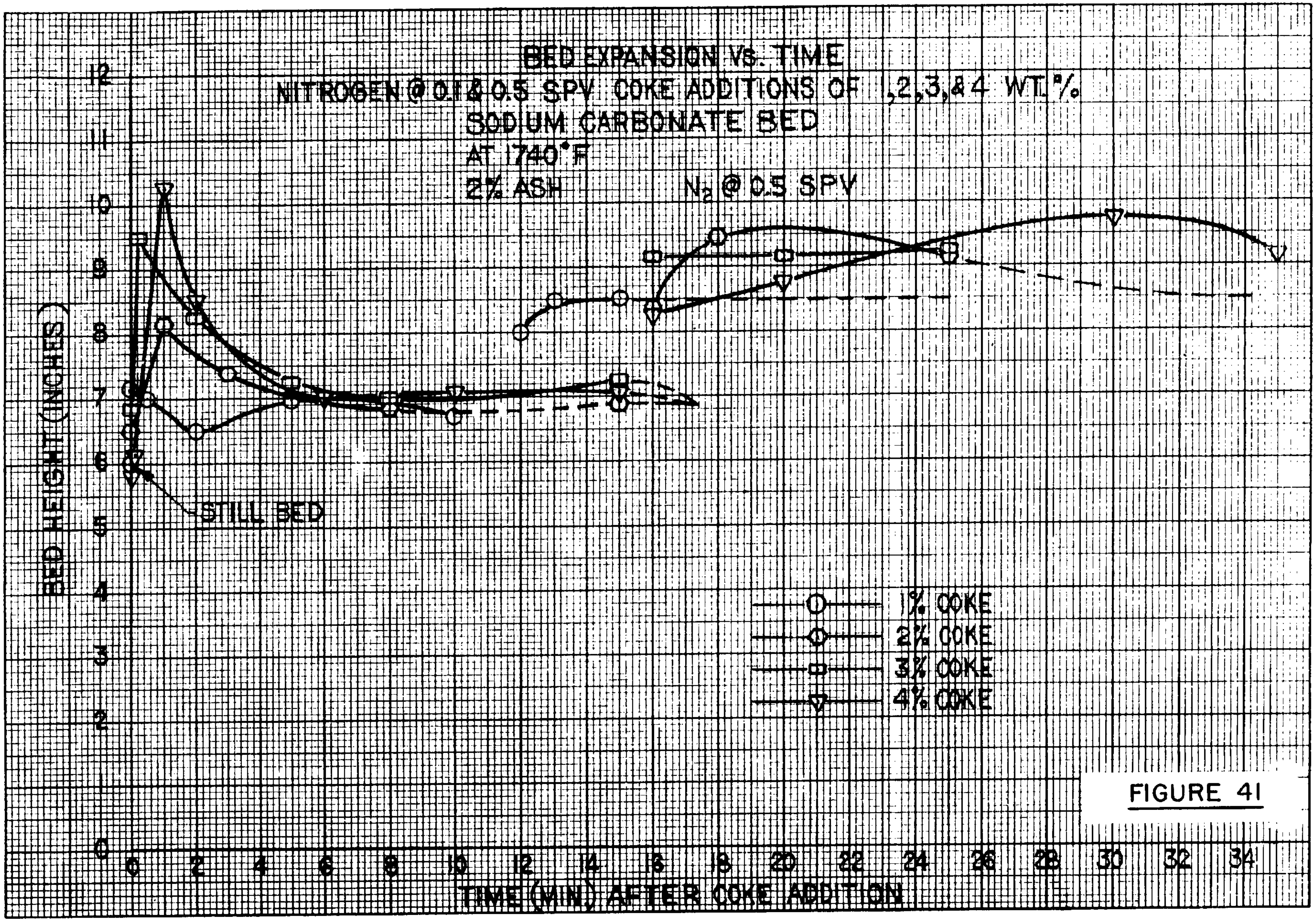

BED EXPANSION Vs. TIME
NITROGEN @ 0.1 & 0.5 SPV COKE ADDITIONS OF 1,2,3,&4 WT.%
SODIUM CARBONATE BED
AT 1740°F
2% ASH
N_2 @ 0.5 SPV
BED HEIGHT (INCHES)
0
2
3
4
5
6
7
8
9
10
11
12
STILL BED
1% COKE
2% COKE
3% COKE
4% COKE
0
2
4
6
8
10
12
14
16
18
20
22
24
26
28
30
32
34
TIME (MIN) AFTER COKE ADDITION

FIGURE 41

The effect of adding 1, 2, and 4 weight percent of coke to a 7-inch bed of molten sodium carbonate under simulated gasification conditions is shown on Figure 42. In general, a rapid expansion of the bed occurred followed by a gradual decline. The decline appears more gradual for the higher percent coke addition. Here again the difficulty of measuring a bubbling bed may explain the difference. Also, the bed may have been pre-conditioned in some manner by the previous addition of 1 and 2 percent coke; although, approximately one hour was allowed between runs to complete gasification.

Figure 43 shows the effect of ash concentration on bed expansion. Ash concentrations of 2, 5, and 8 percent were tested by addition of 4 percent coke under simulated gasification conditions (0.1 fps superficial velocity N_2 and 0.9 fps SPV steam). As can be seen, the ash concentration appears to have little effect on maximum bed expansion. However, it does appear to significantly increase the time for maximum expansion to develop and extend the duration of maximum expansion. It is of interest to note that the maximum bed expansion is nearly 240 percent under the conditions of this test.

Figure 44 provides the results of an experiment to determine bed expansion under conditions simulating continuous operation on coal feed at a rate of 15 lbs. per hr. per cubic foot of quiescent melt. The ash concentration in the carbonate bed was 7 percent at the start of the experiment but probably increased to nearly 8 percent at the conclusion as a result of the continuous coal feed. A 4 percent initial carbon level was obtained by coke addition at the start of the experiment and accounts for the early peak of the plot. Continuous coal feed was started five minutes after the initial coke addition. The plot after 30 or 40 minutes indicates that a bed expansion of about 100 percent could be expected under continuous steady state conditions. Subsequent tests with deeper beds indicate that the percentage of bed expansions decreases with increasing initial bed depth.

b. Simulation Studies

A series of tests on the bed expansion of water, ethylene glycol and glycerine-water mixture (100 cp. viscosity) was completed in a 5-1/2-inch I. D. plastic tower. Figure 45 shows the test apparatus used to determine the bed expansion. An electrical dip

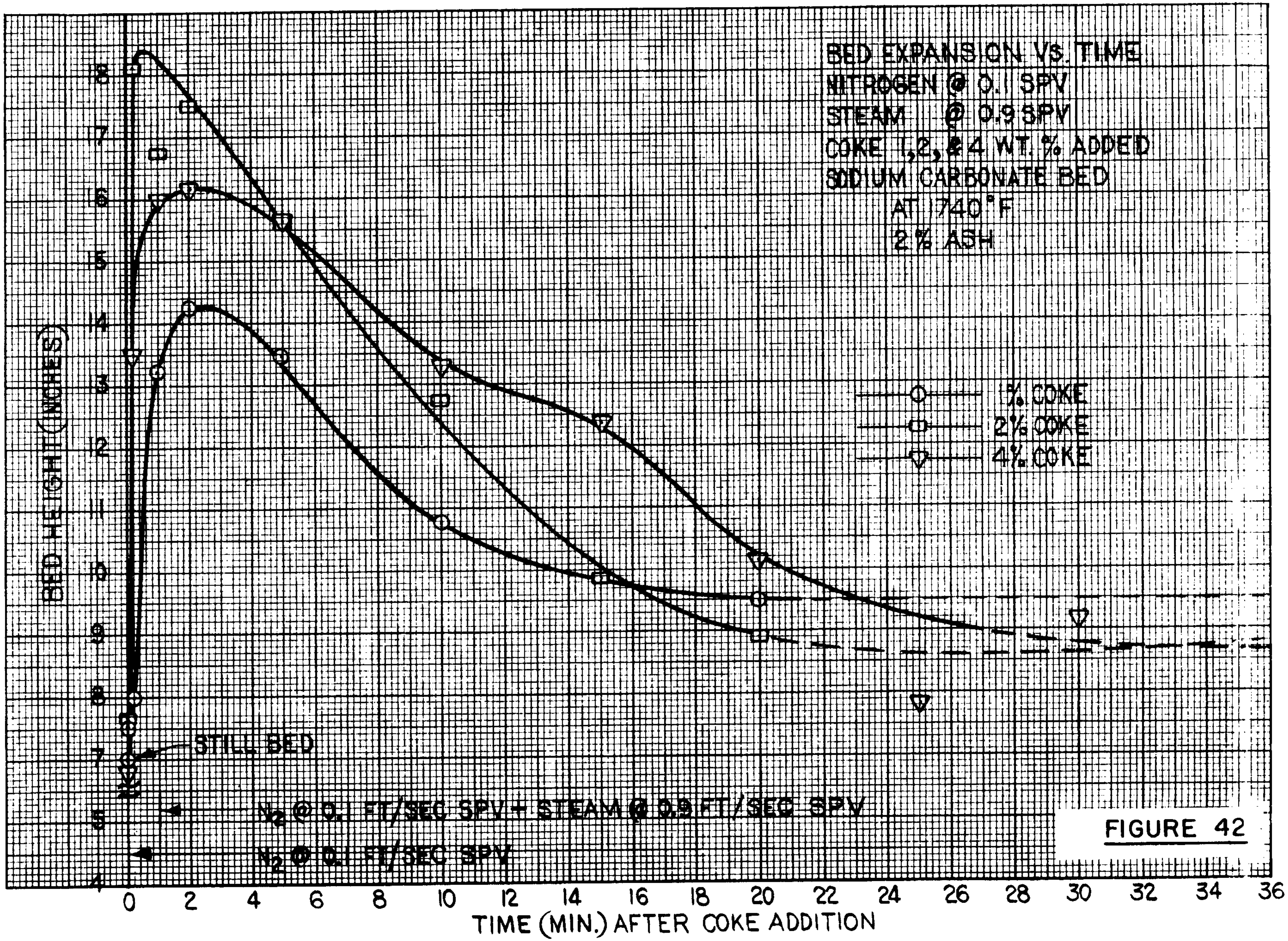

BED EXPANSION VS. TIME
NITROGEN @ 0.1 SPV
STEAM @ 0.9 SPV
COKE 1,2, & 4 WT. % ADDED
SODIUM CARBONATE BED
AT 740°F
2% ASH
1% COKE
2% COKE
4% COKE
STILL BED
N_2 @ 0.1 FT/SEC SPV + STEAM @ 0.9 FT/SEC SPV
N_2 @ 0.1 FT/SEC SPV
BED HEIGHT (INCHES)
18
17
16
15
14
13
12
11
10
9
8
7
6
5
4
0
2
4
6
8
10
12
14
16
18
20
22
24
26
28
30
32
34
36
TIME (MIN.) AFTER COKE ADDITION

FIGURE 42

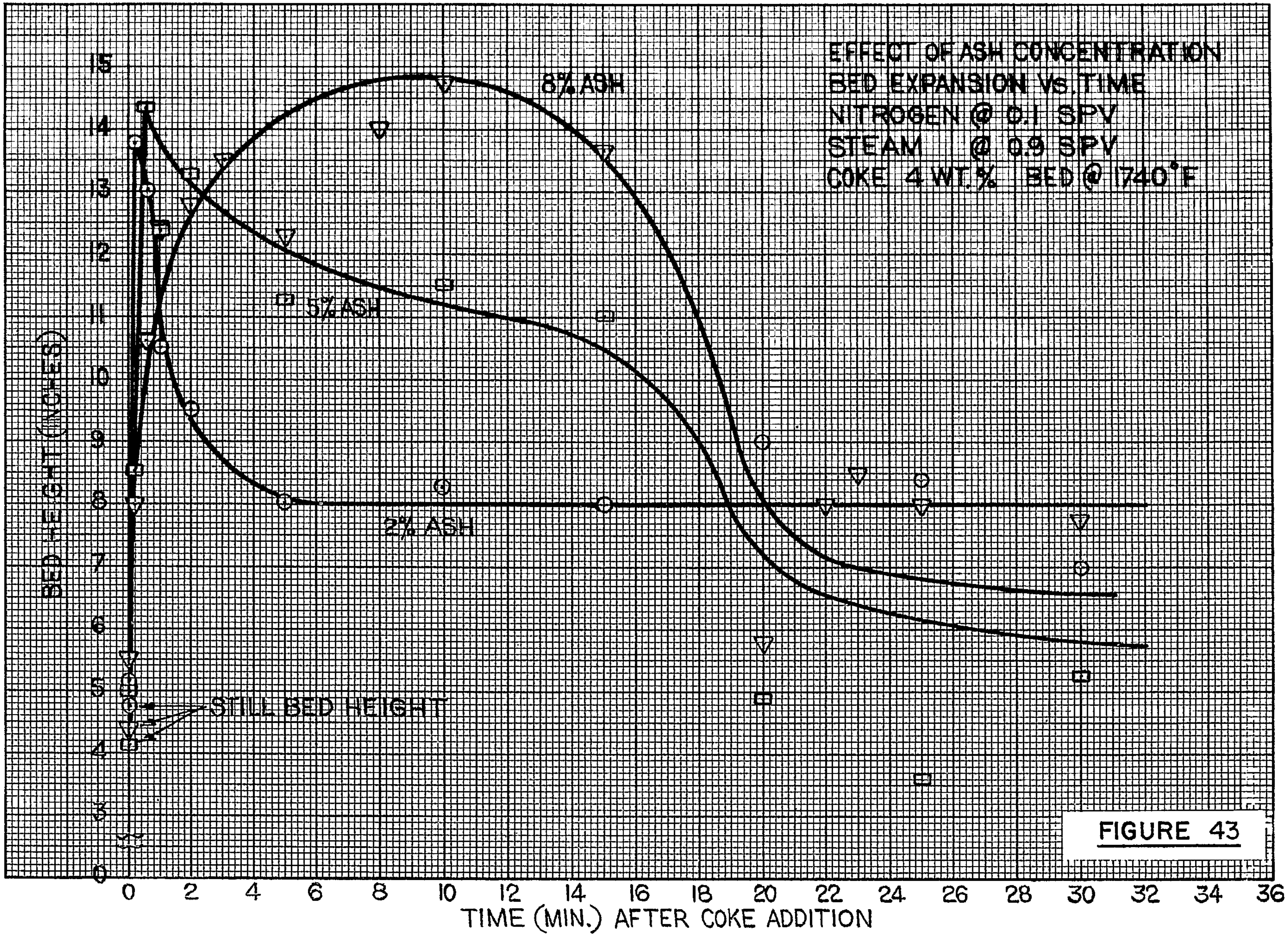

EFFECT OF ASH CONCENTRATION
BED EXPANSION Vs. TIME
NITROGEN @ 0.1 SPV
STEAM @ 0.9 SPV
COKE 4 WT.% BED @ 1740°F
8% ASH
5% ASH
2% ASH
STILL BED HEIGHT
BED HEIGHT (INCHES)
0
3
4
5
6
7
8
9
10
11
12
13
14
15
TIME (MIN.) AFTER COKE ADDITION
0
2
4
6
8
10
12
14
16
18
20
22
24
26
28
30
32
34
36

FIGURE 43

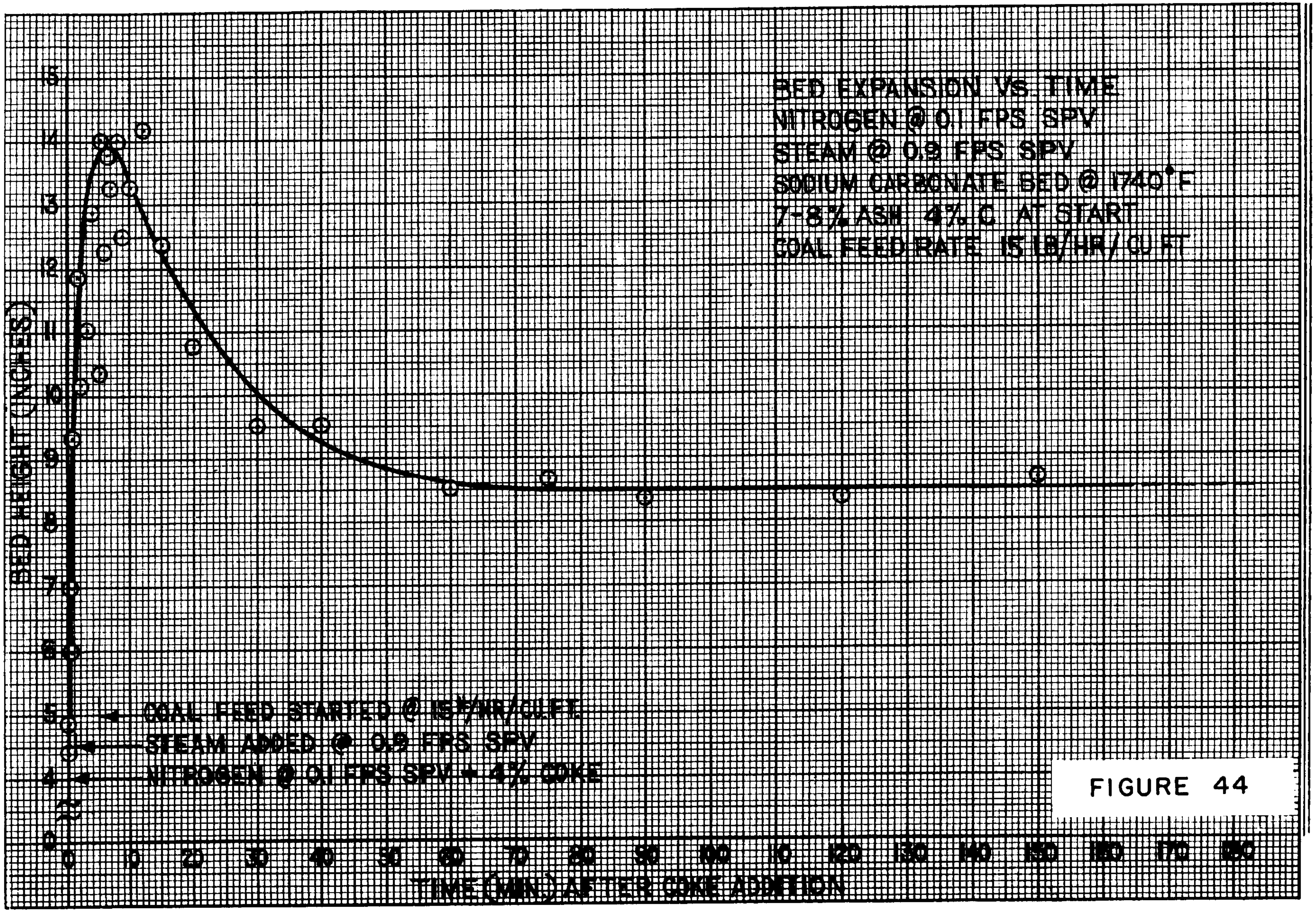

BED EXPANSION Vs TIME
NITROGEN @ 0.1 FPS SPV
STEAM @ 0.9 FPS SPV
SODIUM CARBONATE BED @ 1740°F
7-8% ASH 4% C AT START
COAL FEED RATE 15 LB/HR/CU FT
BED HEIGHT (INCHES)
TIME (MIN) AFTER COKE ADDITION
COAL FEED STARTED @ 15#/HR/CU.FT.
STEAM ADDED @ 0.9 FPS SPV
NITROGEN @ 0.1 FPS SPV + 4% COKE
0
4
5
6
7
8
9
10
11
12
13
14
15
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
160
170
180

FIGURE 44

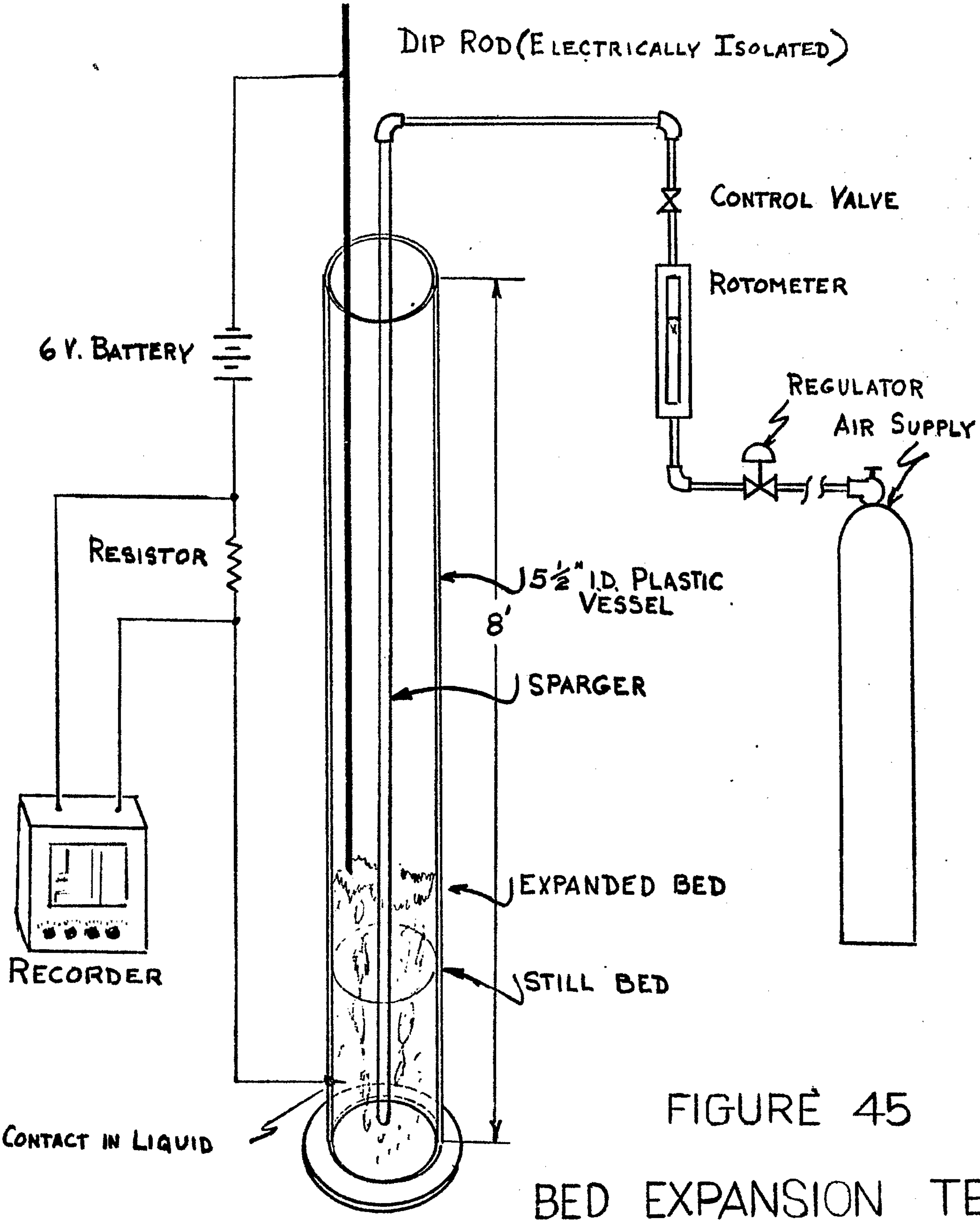


FIGURE 45

BED EXPANSION TEST APPARATUS

stick method using the electrical conductivity of the fluids to complete the circuit was employed. Bed height and expansion were measured by bench mark on the dip stick circuit. In this way, the irregular height of the expanded bed could be measured by comparison of the bed contact pattern recorded on the strip chart. Figures 46 through 48 show the results of these tests. The bed heights shown on the curves are the quiescent (unaerated) bed heights. The range of expansion shown for each bed height was obtained by lowering the dip stick into the expanded bed. The upper curve for each bed height represents the point at which substantial initial contact is made with the bed. The lower curve represents the point where nearly continuous contact is maintained between the dip stick and the bed.

c. Effect of Bed Height

A series of tests was conducted to determine the effect of bed height on melt bed expansion. Figures 49 through 51 show the results of these tests for a one, two, and three foot bed at various bed superficial velocities of carbon dioxide up to three feet per second. These tests were conducted in a 5-3/4 inch I.D. vessel approximately six feet high. The vessel was heated in an electric furnace to approximately 1840°F, producing a melt temperature of 1800°F.

Bed aeration was accomplished by a central sparger consisting of a 1/2 inch pipe inserted from the top of the vessel to within two inches of the bottom. Bed height measurements were made using the electrical dip stick method previously described.

Comparing the three bed heights it will be noted that the nearly continuous contact curve does not substantially change with bed height. However, the upper or initial contact curve varies considerably with bed height. Directionally, this effect was also noted in the other materials similarly tested and previously reported (water, ethylene glycol, 100 cp. glycerine-water mixture). Corresponding to these results at the higher bed levels a significant reduction in splashing and bed loss on the vessel wall was noted.

The following Table 7 briefly summarizes the results of the bed expansion tests. The summary is limited to the results obtained with three foot beds as this is considered the most significant for future application.

FIGURE 46

BED EXPANSION VS AIR SUPERFICIAL VELOCITY

WATER
5-1/2" I.D. TOWER

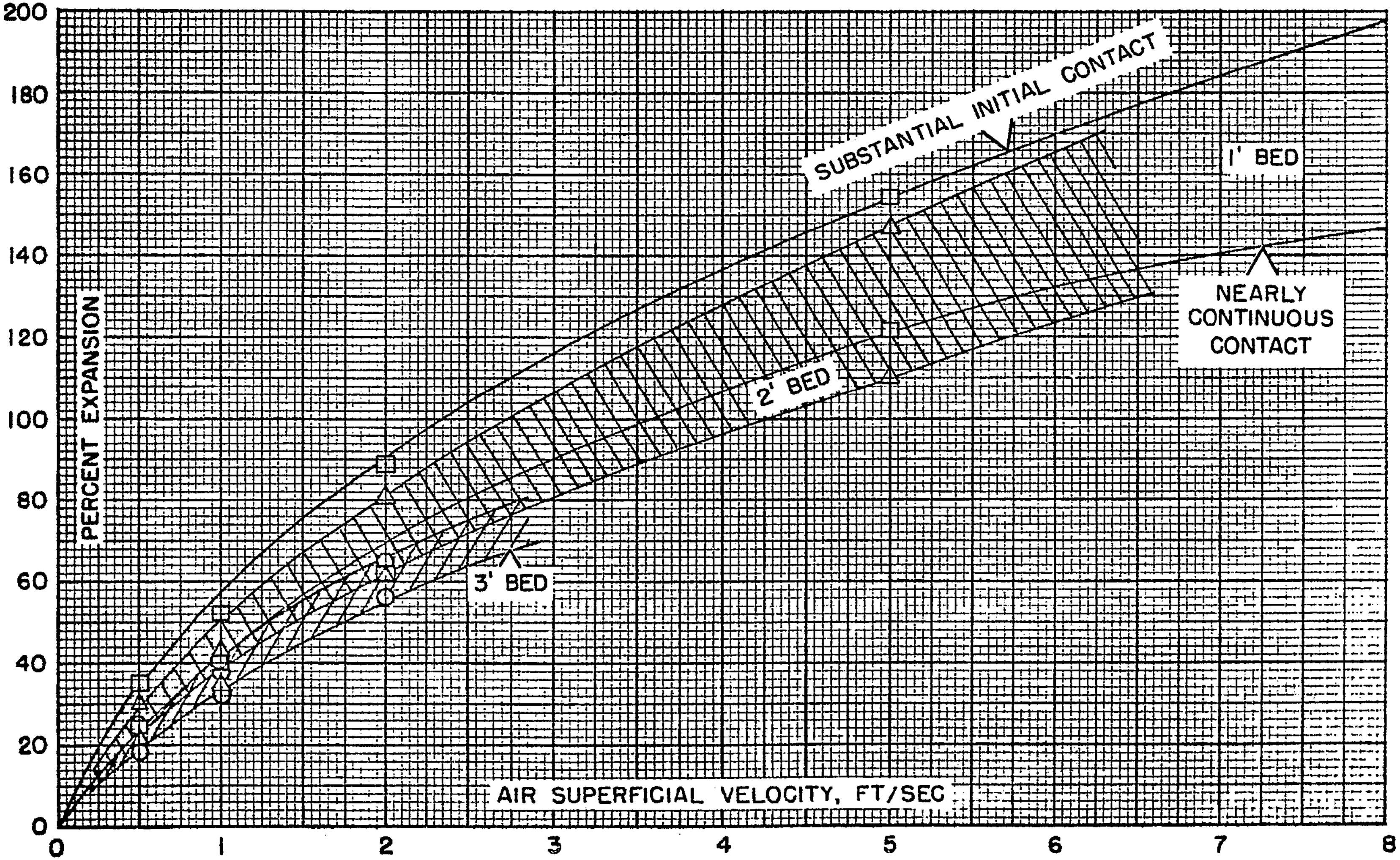


FIGURE 47

BED EXPANSION VS AIR SUPERFICIAL VELOCITY

ETHYLENE GLYCOL
5-1/2" I.D. TOWER

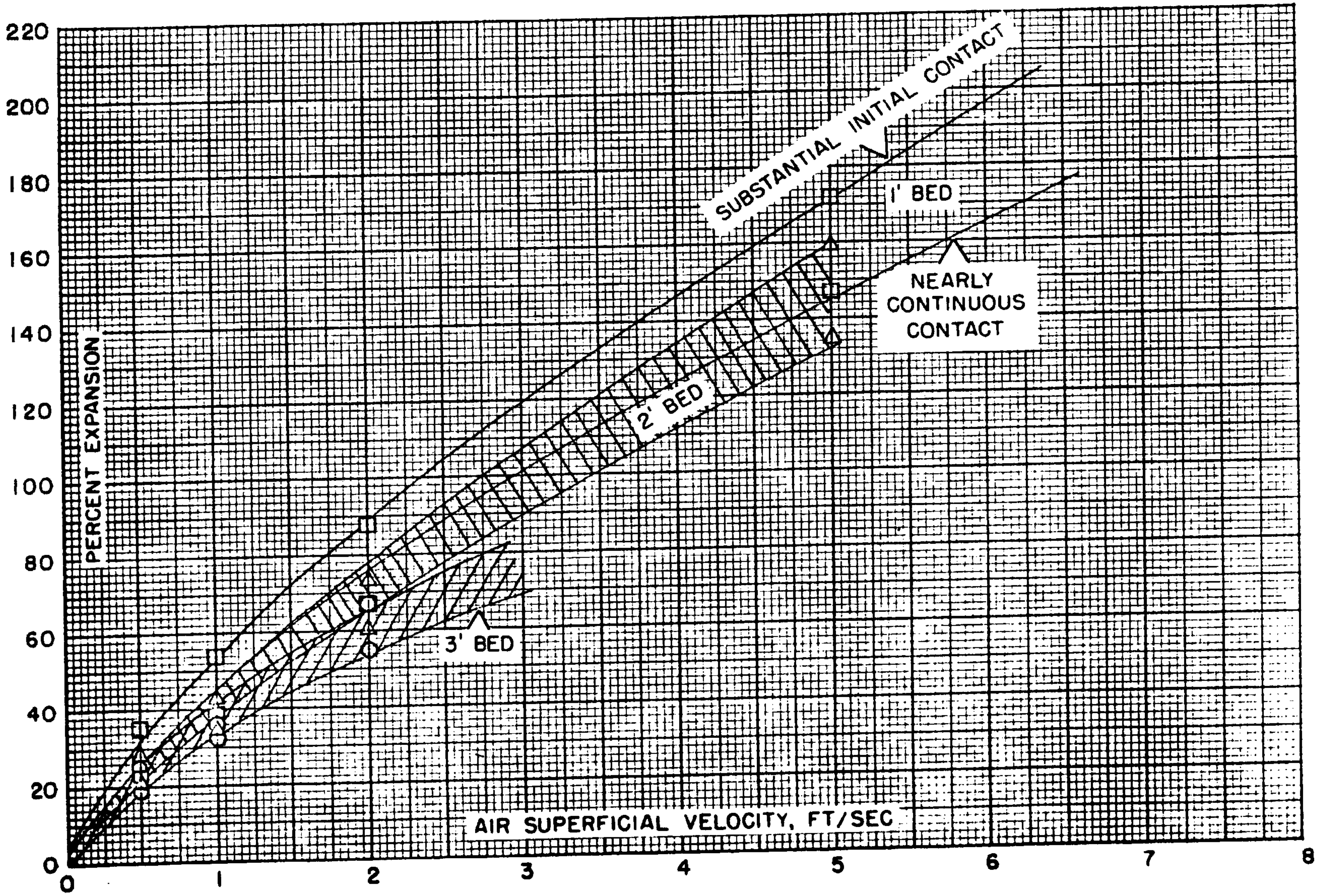


FIGURE 48

BED EXPANSION VS AIR SUPERFICIAL VELOCITY

100 C.P. GLYCERINE – WATER MIXTURE
5-1/2" I.D. TOWER

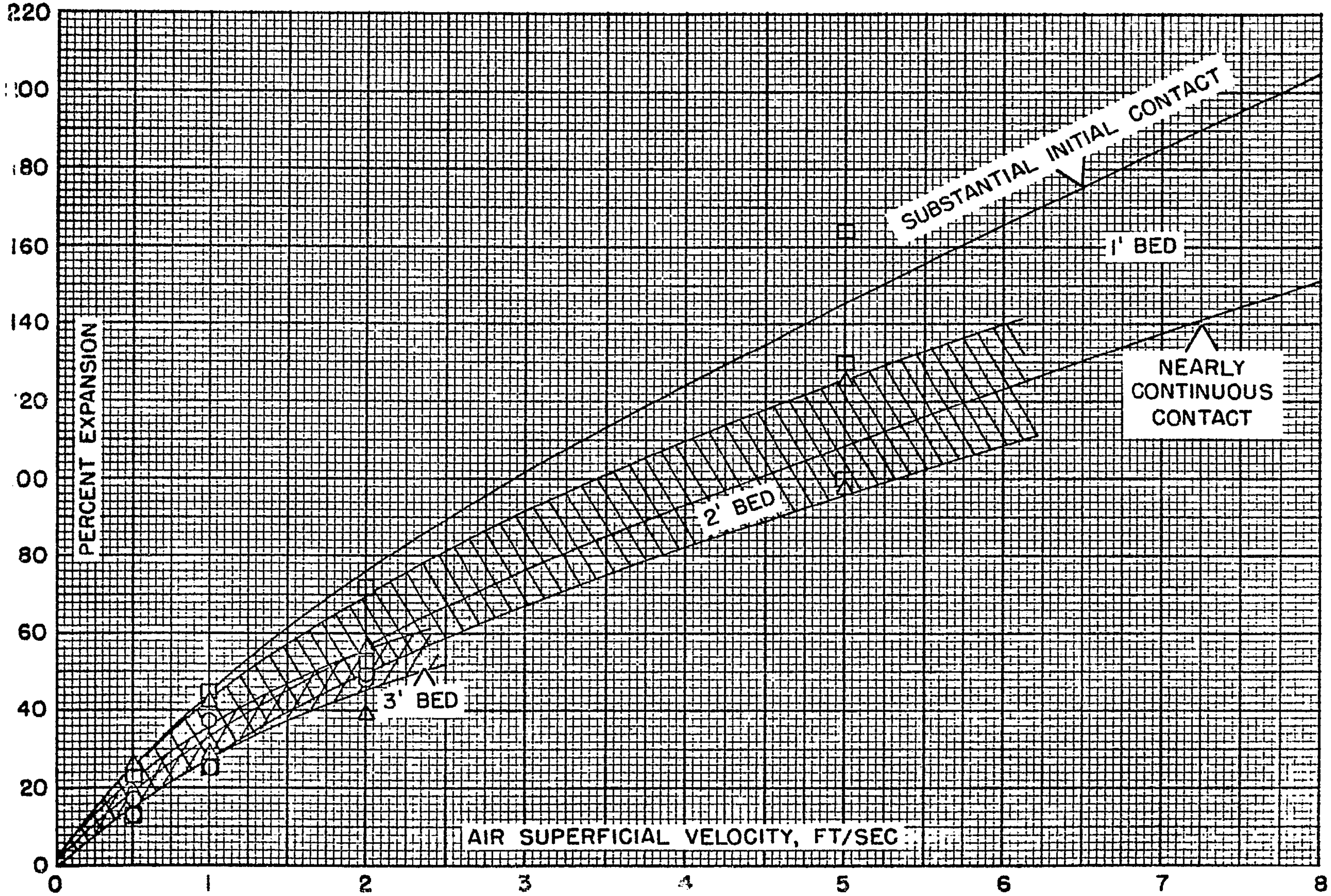


FIGURE 49

BED EXPANSION VS CO_2 SUPERFICIAL VELOCITY

MELT AT 1,800°F 8% ASH
5-3/4" I.D. TOWER
1' BED HEIGHT

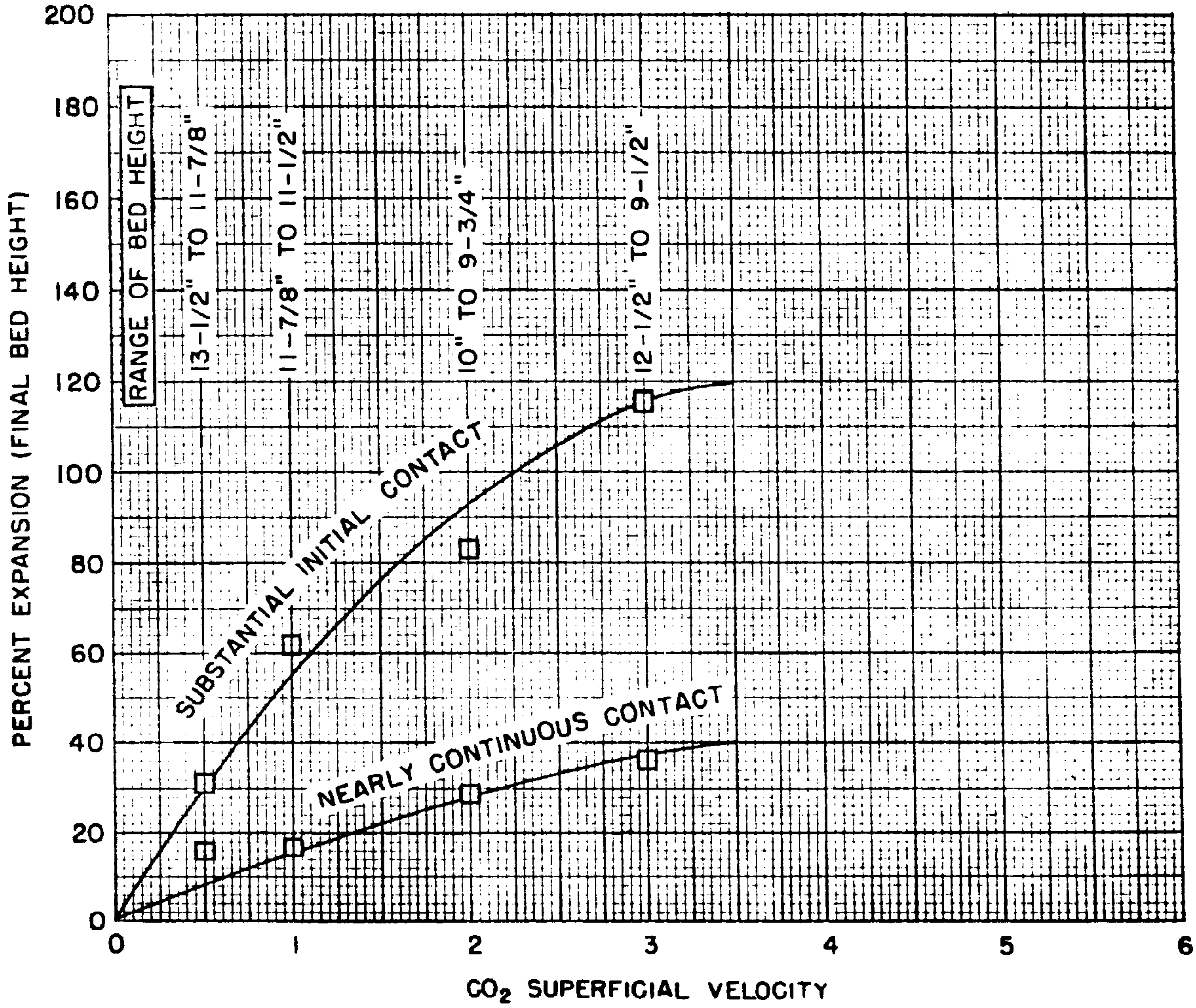


FIGURE 50

BED EXPANSION VS CO_2 SUPERFICIAL VELOCITY

MELT AT 1,800°F 8% ASH
5-3/4" I.D. TOWER
2' BED HEIGHT

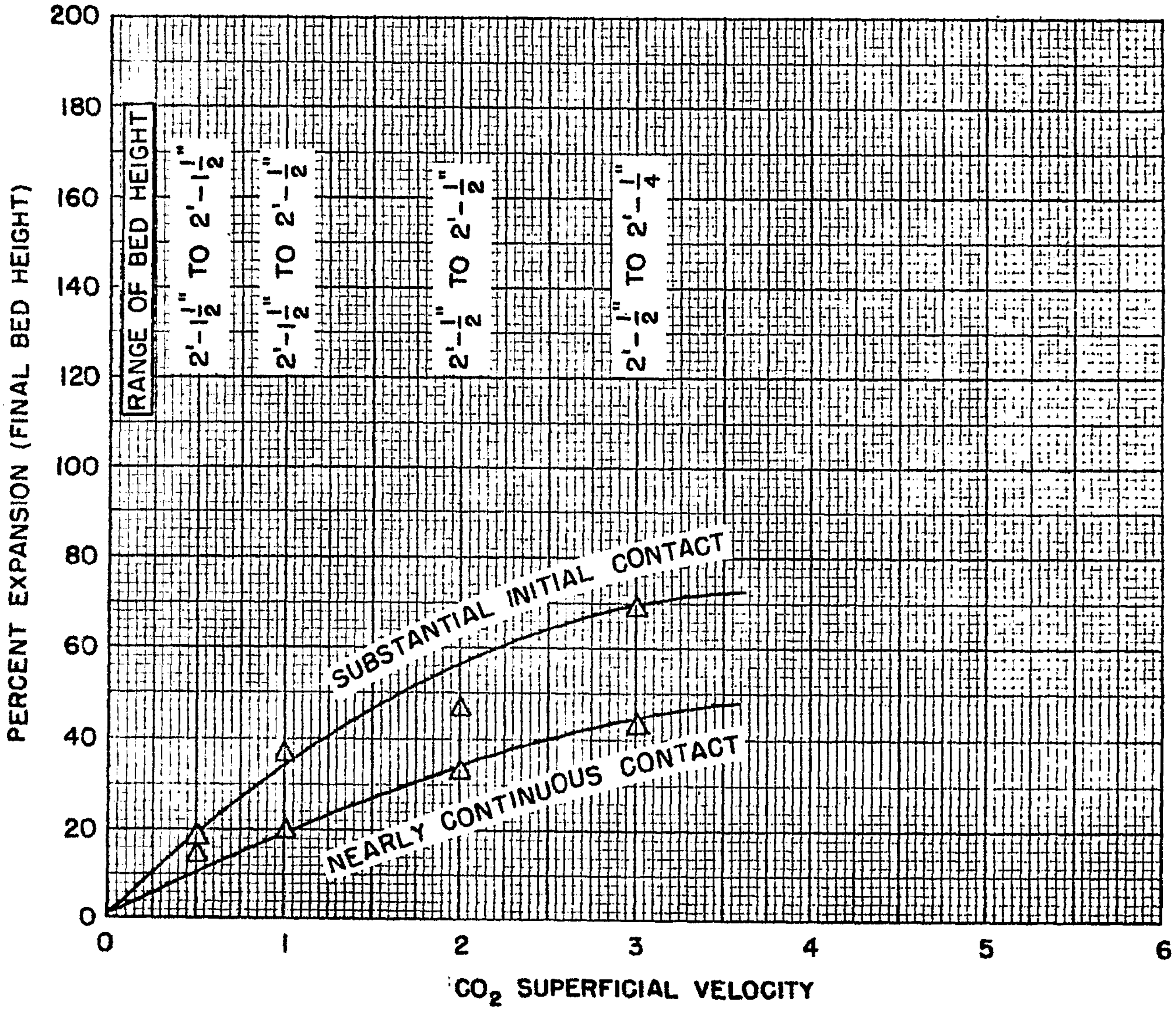


FIGURE 51

BED EXPANSION VS CO_2 SUPERFICIAL VELOCITY

MELT AT 1,800°F 8% ASH
5-3/4" I.D. TOWER
3' BED HEIGHT

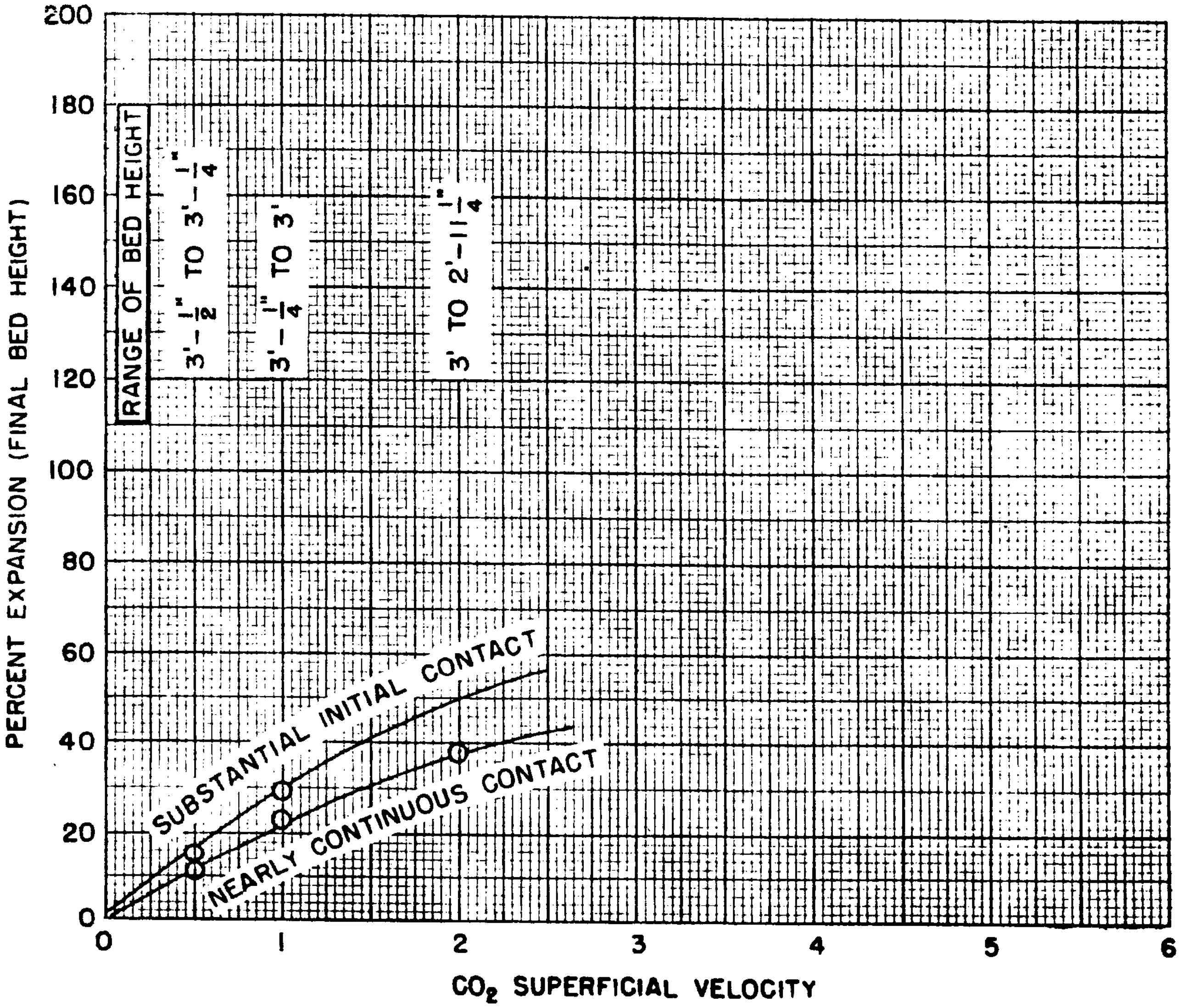


TABLE 7

SUMMARY TABLE

EFFECT OF GAS VELOCITY ON PERCENT BED EXPANSION

MATERIAL	BED SUPERFICIAL VELOCITY 0.5 f/s	 1.0 f/s	 2.0 f/s
Water	18-25	32-42	56-66
Ethylene Glycol	20-25	33-41	55-65
100 cp. Glycerine-Water	15-19	27-36	45-55
Melt 1800°F 8% Ash	11-16	21-29	38-50(E)

* * * * * * * * * *

7. Molten Sodium Carbonate Bed Degasification

Tests have been conducted to determine degasification time for a molten sodium carbonate bed with eight percent ash. Carbon dioxide was used to aerate the bed at superficial velocities up to three feet per second in the test apparatus previously used to determine melt bed expansion.

The electrical dip stick was placed at various heights above the bed and the time was measured from the time the gas supply was shut off until contact was broken between the melt and the dip stick. The results of these tsts are shown on Figures 52 and 53. As can be seen in Figure 53, the degasification time sharply increases below a 1/2-inch gap.

The data indicates the majority of degasification takes place within 10 to 20 seconds regardless of the aeration superficial velocity. Complete degasification would appear to take in the order of two to five minutes based on extension of the data to zero gap. A 1/2-inch gap represents a contamination level of 0.007 cubic feet of gas per cubic feet of melt and was within the acceptable limit set. Thus, a stilling section providing 10 to 20 seconds hold-up would appear adequate to reduce the cross-flow contamination to an acceptable level.

8. Melt Viscosity

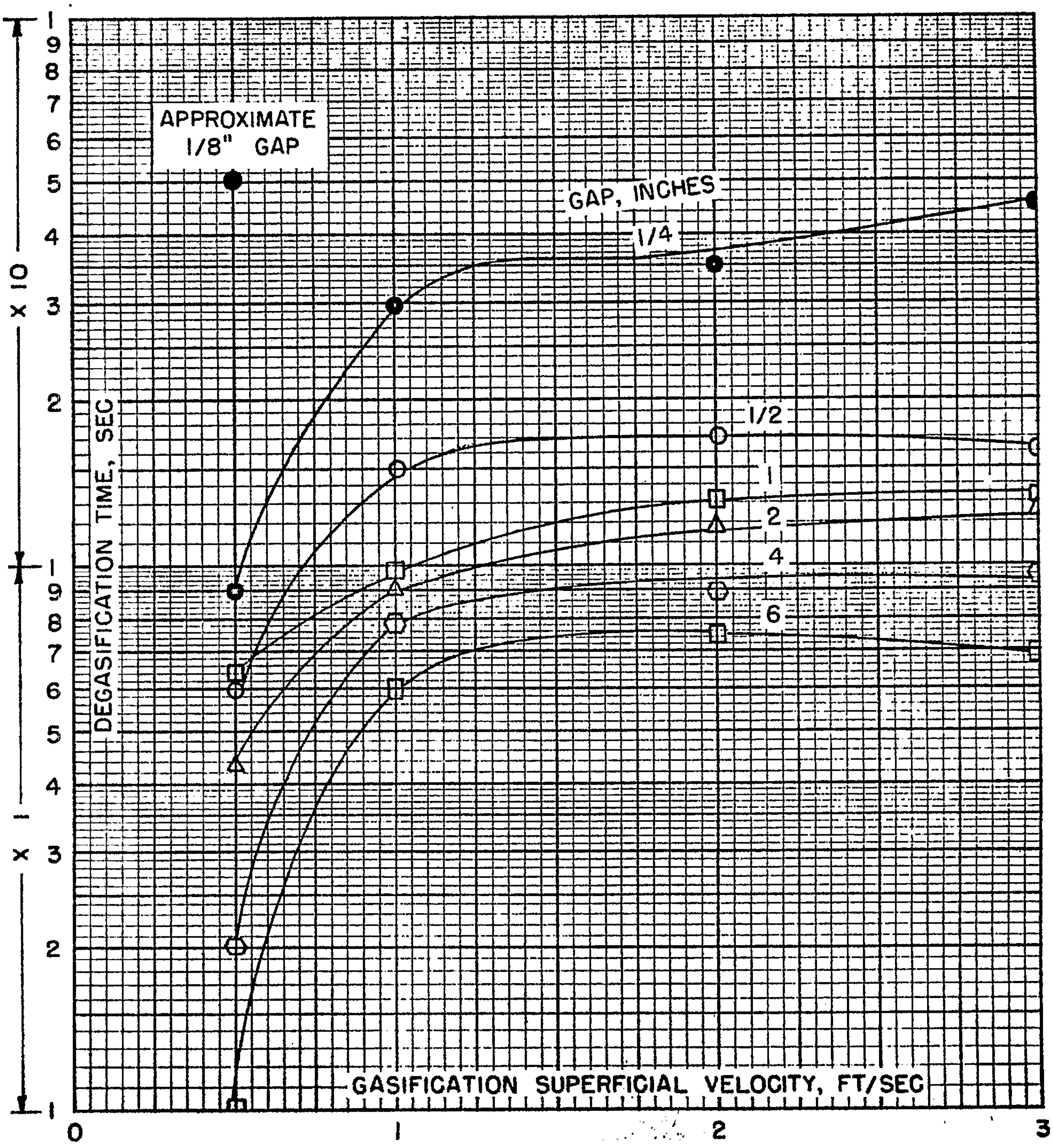
In choosing a material to simulate the Na_2CO_3 melt, a question was raised as to the actual melt viscosity. This in the past has been measured by a Brookfield viscometer, and was found to be approximately 100-120 cp. at 1840°F and 8% ash. However, visual observation of the melt indicated the viscosity might be lower, perhaps more like that of water.

In order to investigate the visocsity problem further, the device in Figure 54 was constructed. This device was filled to a depth of 8-1/2" with a liquid of known viscosity, then the time required for the liquid to flow through the outlet tube was measured. This test was repeated for several liquids including molten Na_2CO_3 + 12% ash; the results being given in Figure 55. From these results it would appear that under conditions of turbulence the viscosity of the molten Na_2CO_3 + ash mixture might be closer to that of water than previously estimated.

FIGURE 52

MOLTEN SALT DEGASIFICATION

1,800°F CO$_2$ GAS



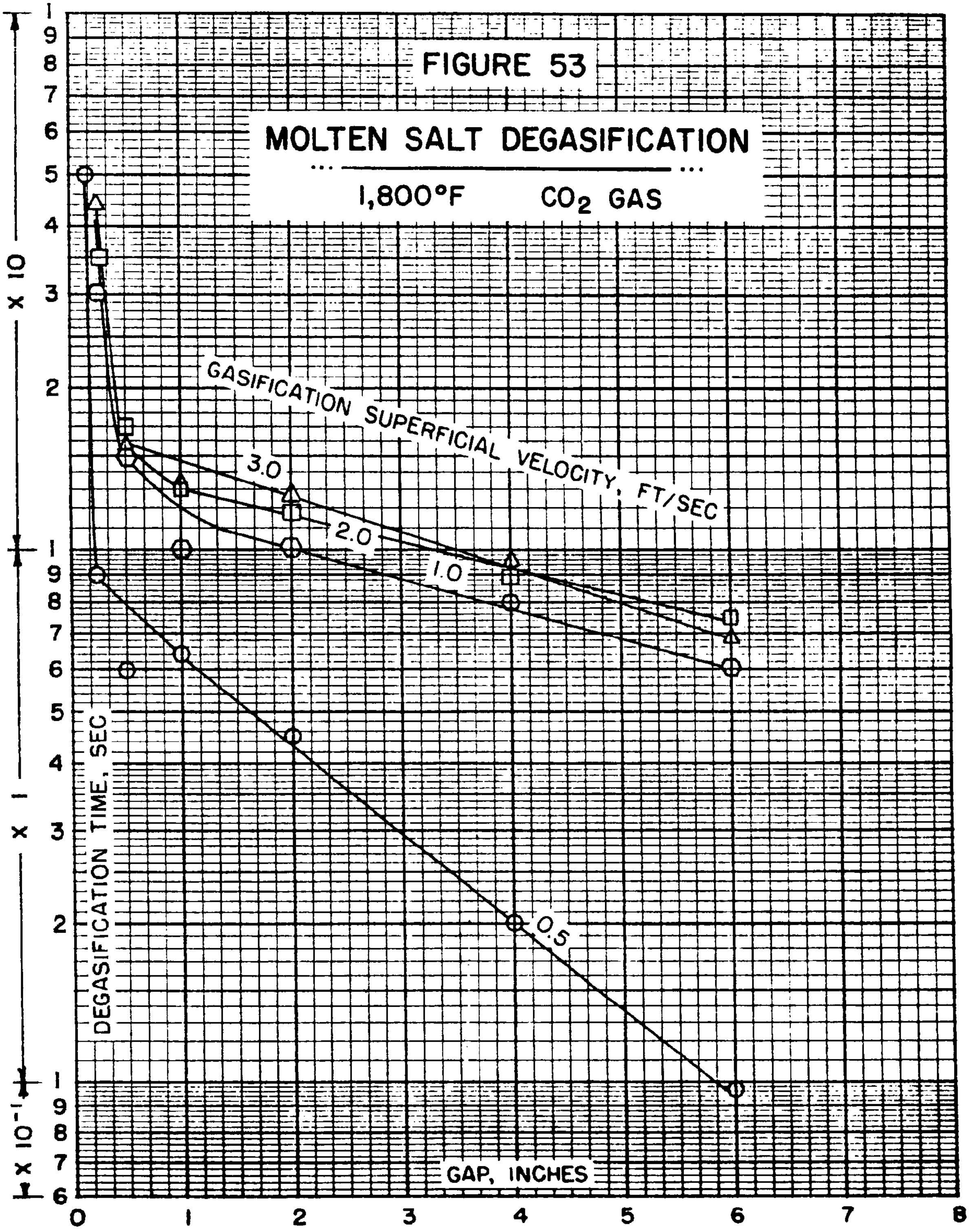
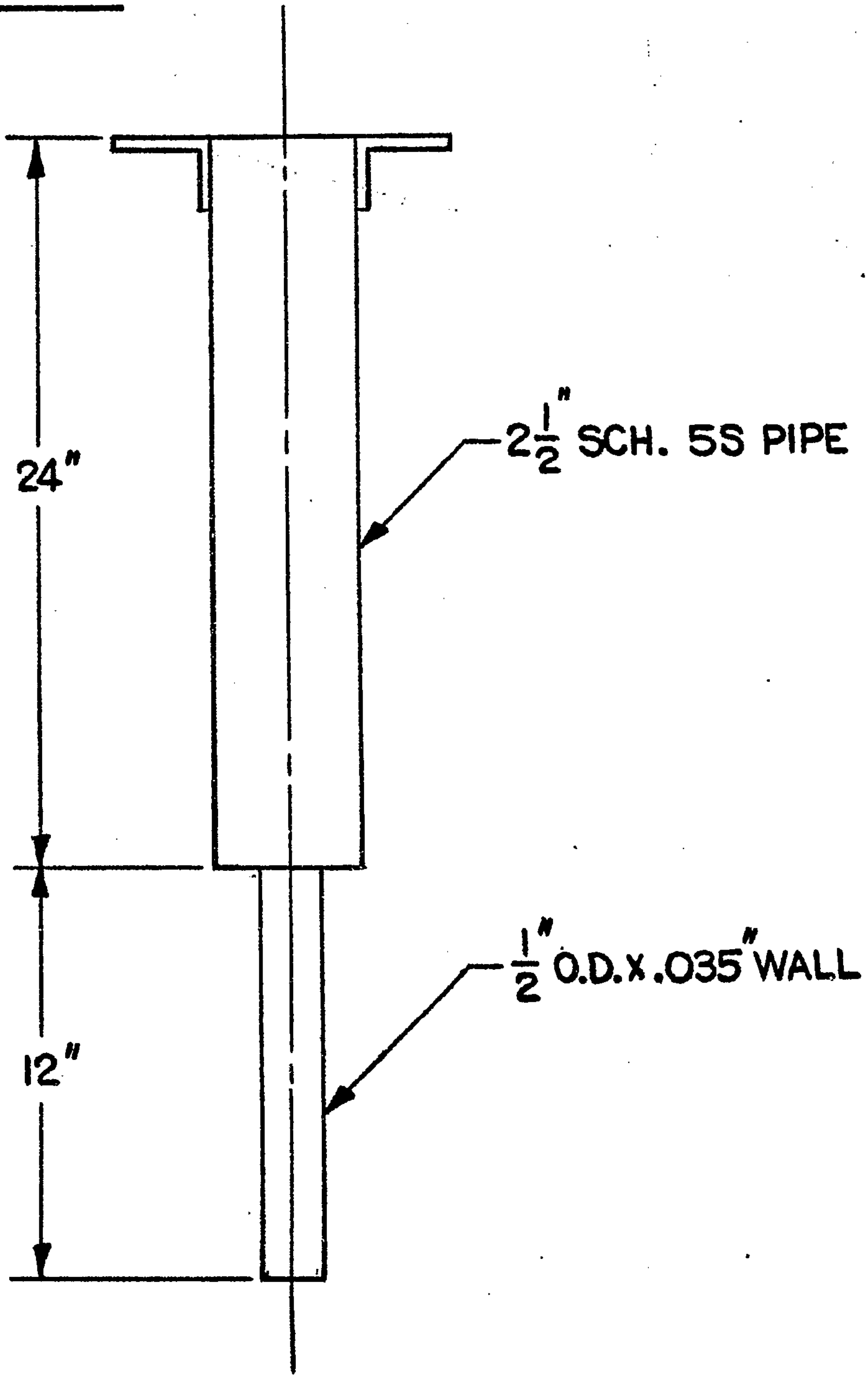


FIGURE 53

MOLTEN SALT DEGASIFICATION

1,800°F CO$_2$ GAS

FIGURE 54 MELT OUTFLOW VESSEL



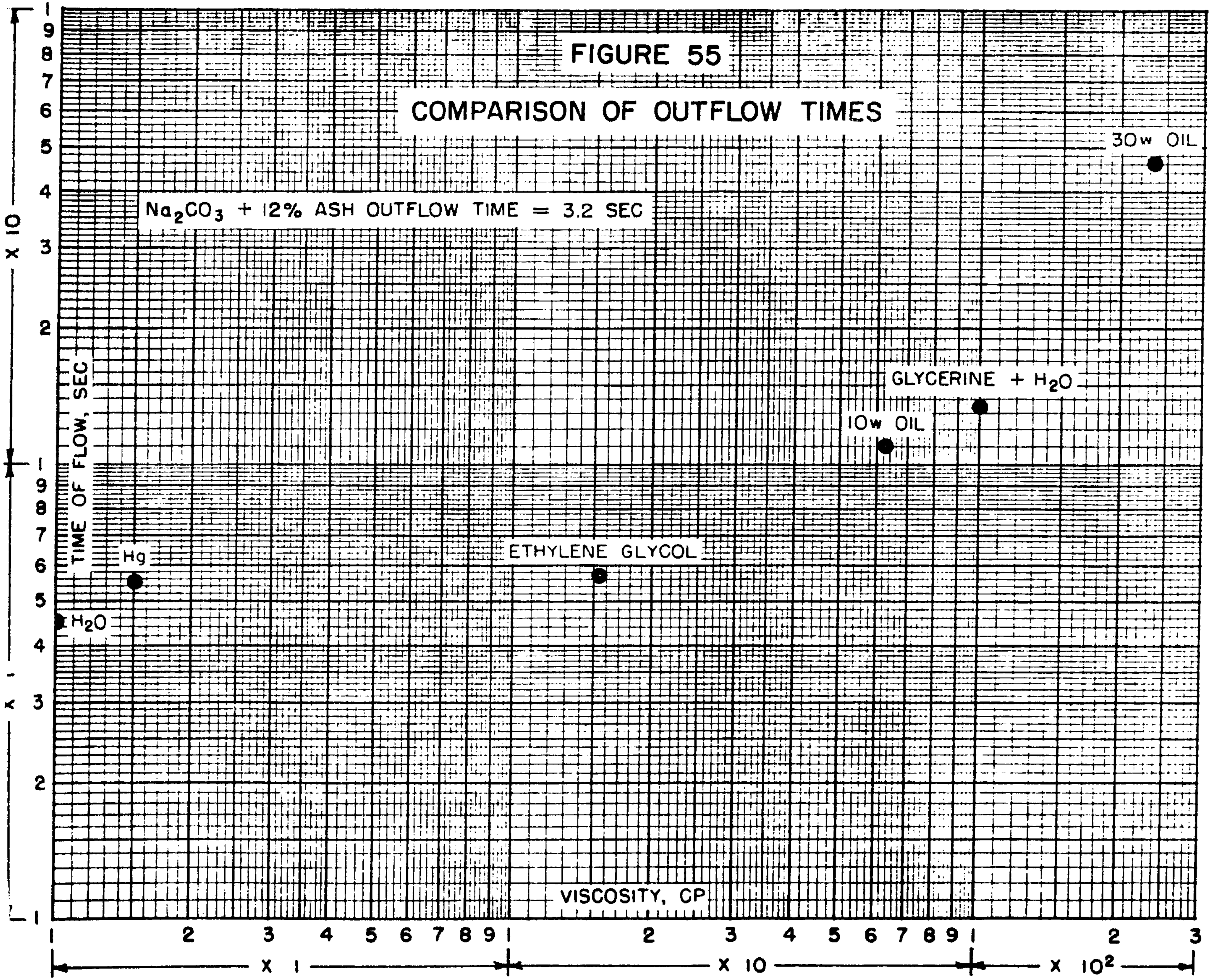


FIGURE 55

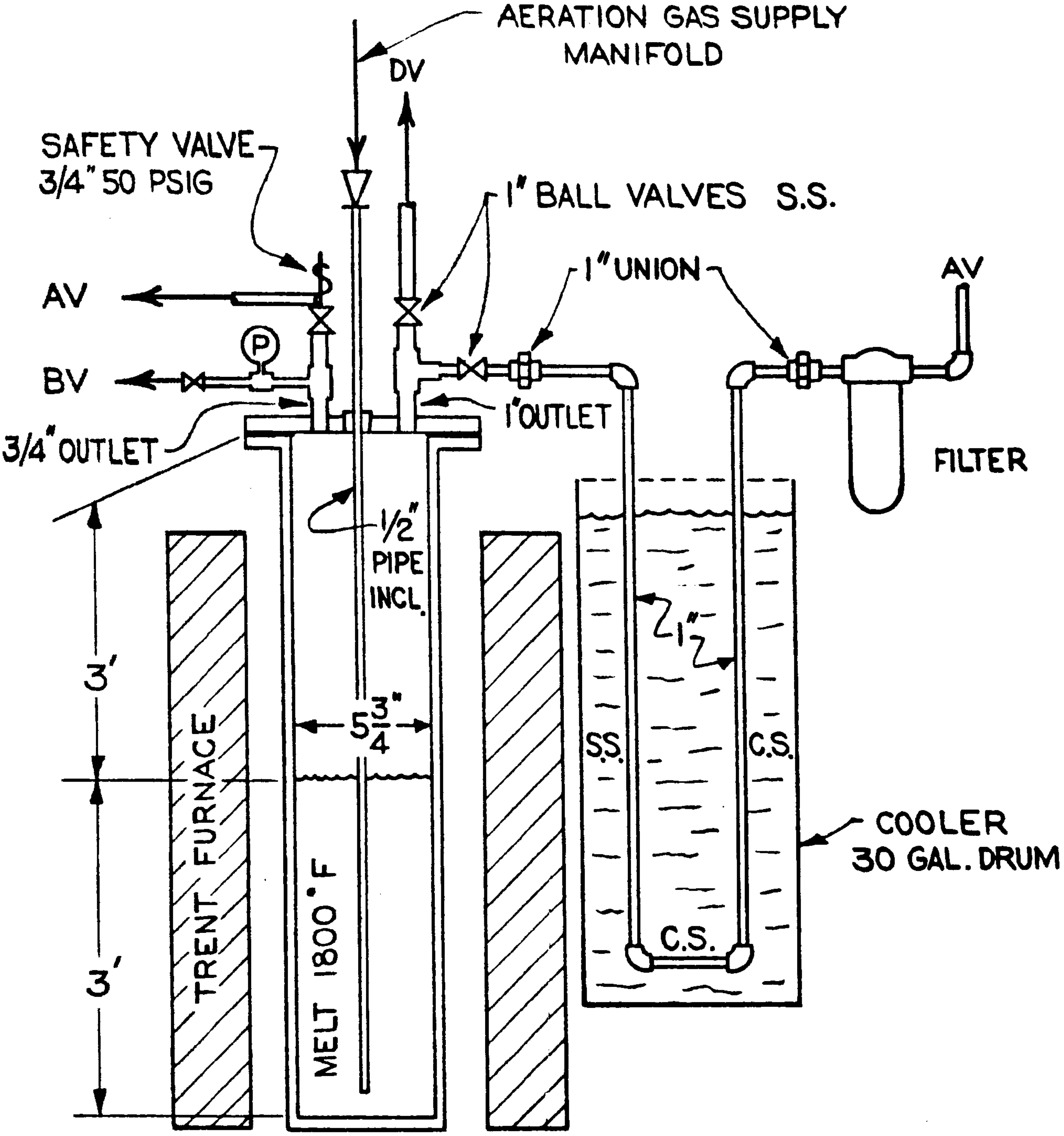
COMPARISON OF OUTFLOW TIMES

9. Salt Carryover

Tests were conducted to determine entrainment in the aeration gas exiting a sodium carbonate melt containing eight percent ash. Carbon dioxide was used as the aeration gas. The tests were carried out in the 5-3/4-inch I. D. vessel shown on Figure 56. Tests were completed for 0.5 feet per second and 1.5 feet per second superficial velocity of the aeration gas. Table 8 shows the results of these tests.

Listed in Table 9 are the analyses of four samples of deposits taken from the exit line cooler and filter. The term "inlet" refers to the piping from the reactor exit to the first bottom elbow of the cooler. The term "bottom" refers to the bottom pipe of the cooler including the two elbows. The outlet line includes the remainder of piping to the filter.

The melt bed contained 8% ash. Analysis shows this ash to contain 38.3% SiO_2. The deposits appeared to be uniformly distributed on the walls of the exit pipes and it would appear from the data that the great majority of carry-over resulted from physical entrainment in the exit gas. Volatile carry-over could not be reasonably predicted by this test.



A.V. ATMOSPHERIC VENT
D.V. DUCT VENT
B.V. BLEED VENT

FIGURE 56
O.C.R. COAL GASIFICATION
CARRYOVER TESTS

TABLE 8

ENTRAINMENT IN AERATION GAS EXITING A SODIUM CARBONATE MELT BED (CO_2 AERATION GAS - 8% ASH IN BED)

3 FOOT BED DEPTH, 5-3/4 INCH I.D. VESSEL

TEST	BED SUPERFICIAL VELOCITY (FT/SEC)	TIME OF TEST (HRS.)	BED TEMP (AVG.) °F	EXIT TEMP (AVG.) °F	COOLER INLET TEMP °F	COOLER EXIT TEMP °F	DEPOSIT IN EXIT LINE WT. (GRAMS)	DEPOSIT IN FILTER WT. (GRAMS.)
	0.5	2.0	1822	715	375	70	6 gms. (White Powder)	Some Evidence Not Measurable (White Powder)
	1.5	2.0	1783	1065	860	240	3 gms. (White Powder)	3 gms. (White Powder)
3	1.5 (2.5' Bed Height)	7.5	1795	960	790	210	Inlet 3.74 gms.* Bottom 3.62 gms.* Outlet 1.6 gms. (White Powder)	3.72 gms.* (White Powder)

*Samples sent for content analysis.

TABLE 9

COMPOSITION OF MATERIAL CARRIED OVERHEAD

FROM

MELT ENTRAINMENT TESTS

LOCATION	DEPOSIT WT.	% Na_2CO_3*	% SiO_2*	% MISC.*
Inlet	3.74 gms.	96.6	1.90	1.5
Bottom	3.62 "	95.9	2.84	1.26
Outlet	1.6 "	92.0	2.18	5.82
Filter	3.72 "	98.2	0.96	0.84
	12.68 gms.			

Aeration Gas - Carbon Dioxide
Bed Height - 2 ft. 6 in.
Bed Superficial Velocity - 2.0 ft./sec.

*(Dry Basis Approximate)