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LIMESTONE INVESTIGATION

FLUIDISED BED CALCINATION OF MT. GAMBIER LIMESTONE

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AUSTRALIAN MINERAL DEVELOPMENT LABORATORIES

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DEPARTMENT OF MINES
SOUTH AUSTRALIA.

LIMESTONE INVESTIGATIONS

FLUIDISED BED CALCINATION OF MT. GAMBIER LIMESTONE

by

J. McE. Hopkins

This Report describes work undertaken by A.M.D.L. as part of a general investigation into the fluidised-bed calcination of Mt. Gambier limestone.

The experimental work was carried out under the general supervision of F.R.Hartley, Chief Chemist

Acknowledgment is made to C. Watts for analyses

Issued by
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ABSTRACT

This report describes the fluidisation and calcination treatment of two samples of Mt. Gambier polyzoal limestone under batch and continuous conditions.

Although high grade lime could be produced under single stage conditions, dust losses were high and attempts to obtain continuous multistage solids transfer were unsuccessful.

1. SUMMARY

Investigations have been carried out on the fluidisation treatment of two samples of Mt. Gambier polyzoal limestone. After crushing to minus 10 mesh this material can be fluidised quite well in a single stage reactor under batch and continuous conditions. With continuous feed higher air flow rates were required to avoid sanding of the minus 10 plus 25 mesh particles in the bed. However attempts to obtain continuous feed conditions in multistage fluid-bed models were unsuccessful, due to fluidisation of the solids in the transfer tubes. Variation in transfer tube diameters and distance from the distributor plates did not overcome this phenomenon in 2.75 inch and 6 inch perspex three stage models.

Attempts to obtain multistage solids transfer in a cross flow type fluid-bed model were also unsuccessful when using countercurrent gas flow.

Batch tests on screen fractions produced from the crushed minus 10 mesh stone indicated that the best size range for fluidisation was minus 10 plus 72 mesh B.S.S. The two samples of limestone investigated comprised an age hardened fine grained sample which had been mined three years previously and a coarse grained freshly mined sample. The crushing characteristics of these two samples were markedly different when crushed to minus 10 mesh, the former giving a sizing of 47 per cent. plus 72 mesh and the latter 80 per cent. plus 72 mesh B.S.S.

Fluid-bed air sizing of the minus 10 mesh stone produced suitable sized feed for the calcination stage but dust losses were high. Optimum conditions occurred for the aged sample when fluidised at an air space velocity of 1.75 ft./sec. for 30 minutes. This gave a product sizing of 89 per cent. plus 100 mesh representing only 58 per cent. of the feed. For the new sample a space velocity of 2.5 ft./sec. and a retention time of 10 minutes gave a product sizing 97 per cent. plus 100 mesh, representing 85 per cent. of the feed.

For good fluidisation of these air sized fractions at an aspect ratio of 1:1 (fluidised), air space velocities of 1.43 and 2.0 ft./sec. were required for the aged and new samples respectively.

Batch calcination tests carried out on these samples in a 3-inch diameter silica, gas-fired furnace at 1000°C produced a high grade lime containing over 96.5 per cent. available CaO within 1 minute of the temperature reaching 1000°C. Dust losses in the furnace were quite high being of the order of 12-15 per cent. of the original minus 10 mesh feed for both samples.

The lime produced was highly reactive and gave high rates of hydration in water.

The overall dust loss in screening and calcining the aged sample was approximately 68 per cent. of the original minus 10 mesh feed. With air sizing instead of screening prior to calcination the overall dust loss would still exceed 60 per cent. of the feed.

For the new sample after air sizing and calcination the dust loss was approximately 28 per cent.

Due to high dust losses and the inability to obtain multistage solids transfer, this type of limestone would not be suitable for calcination by the fluidised bed process.

2. INTRODUCTION

This project was inaugurated in January 1959, to examine whether the fluo-solids technique could be successfully applied to the calcination of high grade South Australian Limestones.

Earlier work carried out in the Metallurgical Section (R.D. 20) on the calcination of Mt. Gambier Limestone, had shown that conventional type kilns would not be suitable for production of high grade lime due to its extreme softness and friability. It was therefore decided to examine the fluidised bed technique for calcining this type of limestone.

3. MATERIAL EXAMINED

3.1 Mt. Gambier Limestone: Aged Sample

This material is a polyzoal limestone composed of fossil sections which appear to be amorphous. The stone is very white in colour, extremely porous and can absorb up to 37 per cent. of its own weight of water and still appear to be relatively dry. The stone contains approximately 98 per cent. $CaCO_3$, is very low in silica, alumina and iron content, and has a bulk specific gravity of 1.3.

The aged sample was obtained from a previous sample used by the Metallurgical Section in 1955, and came from the north and south Sections of Gerricks' Pit, Section 138-Hundred of Blanche, County of Grey, Mt. Gambier. The sample was found to be fine grained and had age-hardened considerably.

3.2 Mt. Gambier Limestone: New Sample

This sample was received in two sealed 44 gallon drums on March 23rd 1959 from Limestone Products Ltd., Mt. Gambier. The stone consisting of 6-inch lumps had been loaded in its moist state straight from the quarry, the drums being kept sealed during transit to Adelaide.

Although of the same origin as the aged sample this stone as freshly mined, contained approximately 5 per cent. moisture, was slightly off-white in colour and coarse grained. It crumbled easily to its natural particle size, these small particles being resistant to further crushing.

The grade of this stone was also 98 per cent. $CaCO_3$.

4. EQUIPMENT

4.1 2.75-inch Diameter Perspex Fluid-Bed Model

This unit was made in flanged interchangeable sections from 2.75-inch internal diameter perspex tube. An air inlet was set at the bottom and a dust collector at the top. Air distribution plates were made from 3/16-inch perspex sheet drilled with 1/32-inch diameter holes at 1/8-inch centres.

The unit could be used to simulate conditions in a single or multistage batch or continuous fluid-bed reactor. Provision was made in each section for manometer tappings for measuring pressure drop across each bed.

4.2 3 inch Diameter Silica Fluid-Bed Reactor

This unit consisted of a 3-inch inside diameter fused silica tube, 30 inches long and fitted with a gas distributor plate approximately 13 inches from the bottom. This plate contained nine silica air domes, each containing 4 x 1/32-inch diameter holes equally spaced for air entry.

The bottom of the tube was sealed except for a 1/4-inch diameter silica tube for air entry. The air for fluidisation was preheated in this 13 inch long section, before passing through the distributor plate.

The whole reactor fitted neatly inside a vertical gas fired furnace fitted with four gas burners.

A chromel-alumel thermocouple was used for temperature control.

4.3 6-inch Diameter Perspex Fluid Bed Model

This unit was similar in design to the smaller 2.75-inch reactor except for the distributor plates, and comprised three multiple stage sections each approximately 2 ft. in height. Two types of air distributor plate were used as follows:

(a) Perspex plate containing 18 brass air nozzles evenly spaced on a circular pattern, with each nozzle containing three 0.054-inch diameter holes drilled through to a common 0.125-inch diameter hole.

Ratio of hole area to plate area = 0.044 sq. inches/sq. inch plate area.

(b) Perspex plate containing 29 brass air nozzles evenly spaced on a circular pattern. Each nozzle contained three 3/32-inch diameter holes drilled through to a common 3/16-inch diameter hole.

Ratio of hole area to plate area = 0.021 sq. inches/sq. inch plate area.

4.4 Perspex Cross-Flow Fluid-Bed Model

This unit was a four compartment fluid-bed reactor, working on the cross-flow principle and is shown in drawing RC-349.

The air distributor plate in each compartment was fitted with 12 brass nozzles having 3 x 3/32-inch diameter holes per nozzle.

Final solids discharge from the reactor was controlled by means of an adjustable weir-slide. The reactor could be set up for either parallel or countercurrent air flow to each compartment.

5. EXPERIMENTAL

5.1 Aged Sample

5.1.1 Fluidisation tests on Sized Screen Fractions

In order to determine the fluidisation characteristics of this material, approximately 20 lb of the sample was crushed to minus 10 mesh in the laboratory jaw crusher and rolls, and the following screen fractions were prepared.

Minus 10 mesh plus 20 mesh
" 20 " " 48 "
" 48 " " 65
" 65 " " 100 "
" 10 " " 65 "

Each screen fraction was subjected to batch fluidisation tests in the 2.75-inch reactor at various aspect ratios and air flow rates. Pressure drop in inches W.G. was plotted against air flow-rate in litres/minute. Any peculiarities in bed behaviour were noted.

5.1.2 Batch Sizing Tests on minus 10 mesh Stone

The fluidisation characteristics of minus 10 mesh material were determined in the 2.75-inch reactor with an aspect ratio of 1/2:1 (static) and then batch fluid-bed sizing tests were carried out at various air flow-rates and fluidisation times. At the end of each test both the coarse and fine products were removed, weighed and submitted for sizing analysis. The test giving the best combination of recovery and grade of plus 100 mesh material was taken as being nearest to the optimum conditions.

5.1.3 Continuous Fluidisation Tests on Two Screen Fractions.

The aim was to determine, from continuous fluidisation tests, dust losses and the optimum screen size fraction to be used as feed to the calcination stage.

Two fractions (-10 plus 65 mesh and - 10 plus 100 mesh) were prepared and continuously fed to the 2.75-inch perspex reactor at various feed rates, giving nominal retention times of 10, 20, 30, 40 and 60 minutes respectively.

The air flow-rates were those which gave good fluidisation conditions with a minimum of sanding. Continuous solids feed was maintained by means of a vibrating spatula feeder.

The time taken to reach equilibrium conditions was assumed to be the desired retention time in each case, and immediately equilibrium conditions had been obtained the bed was shut down. The coarse and fine products were removed, discarded and fluidisation resumed. The coarse feed discharge and the dust fractions were then collected over specified time intervals to determine the dust losses. The percentage dust loss was plotted against retention time.

5.1.4 Batch Calcination Tests

Since earlier work in the Metallurgical Section had shown that the best burning temperature for Mt. Gambier limestone was 1000°C it was decided to adopt this temperature for fluid-bed calcination.

Two calcination runs were carried out in the 3-inch silica furnace, one with minus 10 plus 80 mesh feed and the other with minus 10 plus 65 mesh feed. The procedure for both runs was as follows. The empty reactor was heated to temperature in the gas fired furnace. As soon as the temperature reached 1000°C, the air flow-rate for fluidisation was set to give 100 litres/min in the bed at 1000°C, and the feed sample of 170 g of sized stone introduced to the reactor. Immediately the temperature in the reactor had recovered to 1000°C, sampling of the calcines was started.

Approximately 1.5 g samples were taken from the bed with the aid of a silica tube sampler at time intervals of 0, 2, 5, 10, 20, 30 and 40 minutes respectively. In the first run the samples were placed in 4 oz. sample bottles and submitted to the Analytical Section for total CaO and CO_2 determinations. For the second run the samples were transferred quickly to small stoppered weighing bottles inside a dessicator and immediately analysed for CaO and CO_2. This was done to avoid inaccuracies due to moisture and CO_2 absorption from the atmosphere.

Samples of the dust collected from the top of the furnace were submitted for analysis.

Hydration tests were carried out on the calcines remaining in the furnace after each run to determine the quality of the hydrate produced.

5.2 New Sample

5.2.1 Crushing Tests

Samples of freshly mined stone and stone age-hardened in the open air for 10 weeks were crushed to minus 10 mesh B.S.S. in a jaw crusher and rolls with screening between stages. The samples were then screen sized and the sizes tabulated together with those of the old sample for comparison of their crushing characteristics.

5.2.2 Fluidisation Characteristics in 2.75-inch Model

Batch fluidisation tests were carried out on the minus 10 mesh stone at aspect ratios of 1/4, 1/2 and 1/1 respectively in order to determine the fluidisation characteristics. Air flow-rates in litres/minute were plotted against pressure drop in inches W.G.

5.2.3 Continuous Fluid-Bed Sizing Tests

The minus 10 mesh crushed stone was continuously fed to single stage fluidising equipment using various air flow rates

The air space velocity was varied from 2.10 - 3.0 ft/sec. and nominal solids retention time was maintained at approximately 9-10 minutes.

Sizing analyses were carried out on the air-sized stone and dust fractions.

The feed tube was set at one inch above the distributor plate and the overflow tube was adjusted to give a bed aspect ratio of 1:1 when fluidised.

5.2.4 Batch Calcination of air-sized Minus 10 mesh Fraction

The feed material used for the calcination test had been previously air-sized at a space velocity of 2.5 ft./sec. and a nominal retention time of nine minutes. This fraction was 96-97 per cent. plus 100 mesh and contained only 5 per cent. minus 72 plus 100 mesh material.

The procedure adopted in this test was different from previous tests in that the furnace charge of 170 g was placed in the reactor right from the start. This enabled the stone to preheat gradually without subjecting it to sudden thermal shock as previously. The cold air flow-rate was adjusted to give 2.0 ft./sec. space velocity at 1000°C in the bed. Dust losses were determined.

Samples of the calcined stone were taken at zero, 5 and 10 minutes after reaching 1000°C.

A rate of hydration test was carried out on the calcined product.

5.2.5 Multistage Fluidisation: 2.75-inch Model

The perspex reactor was set up as a 3-stage unit with the transfer tubes adjusted to give a bed aspect ratio of 1:1 when fluidised in each stage. The height of the feed tube above the distribution plate in each stage was varied from 1/2" to 2" during the tests.

The procedure adopted generally was to fill each stage with the requisite quantity of air sized stone, then to commence fluidisation with an air space velocity of 2.0 ft./sec and finally start continuous feed to give a nominal retention time of approximately 10 minutes in each stage.

5.2.6 Multistage Fluidisation: 6-inch Model

This consisted of tests using the 6-inch perspex reactor with 3 stages and material with a size range of minus 10 plus 100 mesh.

Firstly two types of air distributor plate described earlier were subjected to pressure drop vs air flow-rate tests to determine the most suitable plate to be used in the continuous tests.

A batch test in one stage was run to determine the fluidisation conditions required prior to continuous multistage testing.

The 6-inch perspex reactor was set up as a 3 stage unit, each stage having been filled manually to give an aspect ratio of 1:1 when fluidised.

11/16 inch inside diameter transfer tubes were used initially, followed by 7/16 inch diameter tubes.

5.2.7 Fluidisation Tests in Cross-Flow Model

Initially one compartment of the reactor was sealed off from the next and filled to the 3-inch level with minus 10 plus 100 mesh stone. Batch fluidisation characteristics were then determined for this material over a wide range of air flow-rates.

Continuous tests were then attempted in this reactor for both countercurrent and parallel air flow. For countercurrent air-solids flow the solids discharge from No.4 cell was connected to an air-lock receiving bottle. While investigating the operation of the reactor, solids were fed manually through a funnel above No.1 cell. The tail of this funnel extended into the stone bed to within 2 inches of the distributor plate. Transfer port openings were varied from 3/16 inch to 3/8 inch during the test.

For parallel air flow conditions a separate air supply was connected to the bottom of each cell and feed to No.1 cell was maintained by means of a Van Gelder vibrating feeder. The maximum feed rate, total air flow-rate and pressure drops obtained in each cell were noted. For this test the baffle openings were set at 3/16 inch.

6. RESULTS

6.1 Aged Sample

6.1.1 Fluidisation Tests on Sized Screen Fractions

A typical sizing of this stone after crushing to minus 10 mesh is given below.

Mesh Tyler	Cum Weight %
+ 10	1.48
+ 14	8.72
+ 20	17.08
+ 28	24.96
+ 35	32.52
+ 48	34.60
+ 65	46.86
+ 100	55.30
+ 150	64.06
+ 200	74.88
- 200	25.12
	100.00

The pressure drop across one of the 2.75-inch diameter perspex distributor plates was determined at various air flow rates. Results were as follows:

Air Flow: Litres/min	Pressure Drop: Inches W.G.
20	0.04
60	0.30
100	0.41
200	1.40

The experimental figures obtained from fluidising tests using the five sized screen fractions are shown plotted in Figures 1-5 inclusive. The zones marked on the figures indicate the condition of the bed over the air flow-rates shown. The boiling zone is that zone where all the solids are suspended and in motion.

The aspect ratios or $^{L}/D$ ratios indicated in the figures refer to the ratio of height of solids to diameter of solids in the bed when stationary.

For the minus 10 plus 20 mesh fraction, the bed was too porous for good fluidisation and bad slugging occurred with aspect ratios greater than 0.25:1. High air flow-rates were needed but dust losses were negligible.

For the minus 20 plus 48 mesh fraction good fluidisation was observed up to an aspect ratio of 1:1, with dust losses occurring in the violent region. At higher aspect ratios bad slugging occurred. The optimum aspect ratio was found to be 0.5:1.

The minus 48 plus 65 mesh fraction caused channelling before the onset of fluidisation, and high dust losses were encountered. No slugging occurred with this fraction and the best aspect ratio was found to be 0.5:1

The minus 65 plus 100 mesh fraction was worse than the previous fraction, in that bad channelling occurred simultaneously with high dust loss before complete fluidisation was obtained. The minus 10 plus 65 mesh fraction was found to fluidise quite well with little dust loss, up to an aspect ratio of 0.5:1. At higher aspect ratios very high air flow-rates were required for complete fluidisation and dust losses from the top of the bed were high. The optimum aspect ratio was found to be 0.5:1 and the dust losses varied from 2.4 per cent. in the first 10 minutes of fluidisation to 4.2 per cent. after 60 minutes. Although this dust loss is small, the minus 10 plus 65 mesh fraction presented only 47 per cent. of the original minus 10 mesh feed.

Figure 6 indicates the percentage dust losses obtained up to a total fluidisation time of 60 minutes.

6.1.2 Batch Sizing Tests on Minus 10 mesh Stone

The pressure drop vs. air flow-rate figures obtained in the fluidisation of this fraction are shown plotted in Figure 7.

Results of the batch fluid-bed sizing tests are shown in Tables 1-9 inclusive. From the sizing results no clean-cut size fraction can be produced by air sizing. The size fraction giving the best combined recovery and grade of plus 100 mesh stone occurred with a space velocity of 1.75 ft./sec. for a nominal retention time of 30 minutes and giving an overall dust loss of 42.4 per cent. This sized fraction contained 89.0 per cent. plus 100 mesh and 96.8 per cent. plus 200 mesh respectively, giving a recovery of 92.5 per cent. of the original plus 100 mesh and 85.7 per cent. of the original plus 150 mesh material in the minus 10 mesh feed. It will be noted from the sizings given in the tables that some attritioning of the coarser feed fractions occurred during fluidisation.

Table 1

Batch Sizing Tests: 2.75-Inch Reactor

Aspect Ratio 0.5:1 (Static Bed)   Material - Minus 10 mesh
Air Flow Rate 72 litres/min   Space Velocity - 1.0 ft/sec

Bed Charge				
Weight g	Weight Loss g	Weight loss %	Pressure drop inches W.G.	Fluidisation Time Mins
155	-	-	1.55	0
139	16	10.3	1.50	5
124.8	30.2	19.5	1.40	10
119	36.0	23.2	1.35	15
113	42.0	27.1	1.30	30
111.8	43.2	27.8	1.25	45
109.8	45.2	29.2	1.20	60
108.0	47.0	30.3	1.20	90

Material Balance

Mesh Tyler	Feed % Cum 100	Size Stone % Cum 69.7	Dust % Cum 30.3
+ 10	1.48	2.6	
28	17.08	23.5	
35	24.96	34.8	
65	46.86	65.5	0.5
100	55.30	75.0	0.75
150	64.06	85.2	1.00
200	74.88	94.8	10.00
-200	25.12	5.2	90.00
	100.00	100.0	100.00

When completely fluidised the bed expansion was 3.5 inches with a few coarse particles remaining stationary on the bottom of the bed. After 60 minutes fluidisation time only 76 per cent. of the bed remained fluidised.

Calculations from the material balance indicated that the following amounts were removed during the run.

22%	of the	- 65 + 100	mesh stone
29.6%	" "	-100 + 200	" "
86.0%	" "	-200	" "

Table 2

Batch Sizing Tests: 2.75-inch Reactor

Aspect Ratio 0.5:1 (Static Bed)   Material - Minus 10 mesh
Air Flow Rate 88.2 litres/min   Space Velocity - 1.25 ft/sec

Bed Charge				
Weigh g	Weight Loss g	Weight Loss %	Pressure drop inches W.G.	Fluidisation Time Mins
155			1.8	0
			1.7	5
			1.42	20
100	54.5	35.1	1.40	30

Material Balance

Mesh Tyler	Feed % Cum 100	Size Stone % Cum 69.7	Dust % Cum 30.3
+ 10	1.48	2.9	
28	17.08	21.5	
35	32.52	42.4	
65	46.86	64.5	0.6
100	55.30	75.4	0.8
150	64.06	86.9	2.2
200	74.88	95.8	19.4
-200	25.12	4.2	80.6

The bed expansion was 4.5 inches when completely fluidised and the bed remained fluidised throughout the whole test. Dust loss was almost negligible after 20 minutes fluidisation.

Calculations from the material balance indicated that the following amounts were removed during the run.

16.3% of the - 65 + 100 mesh stone
32.4% " " - 100 + 200 " "
89.2% " " - 200 " "

Table 3

Batch Sizing Tests: 2.75-Inch Reactor

Aspect Ratio 0.5:1(Static bed) Material - Minus 10 mesh
Air Flow Rate 88.2 litres/min. Space Velocity - 1.25 ft/sec

Bed Charge				
Weight g	Weight loss g	Weight Loss %	Pressure drop inches W.G	Fluidisation Time Mins
155	-	-	1.8	0
			1.6	5
			1.42	10
107.0	48.0	30.9	1.40	15

Material Balance

Mesh Tyler	Feed % Cum	Size Stone % Cum	Dust Frac. % Cum
	100	64.1	30.9
10	1.48	4.4	
28	17.08	23.5	
35	32.52	44.4	
65	46.86	64.7	0.5
100	55.30	75.1	0.75
150	64.06	86.2	1.75
200	74.88	95.1	14.05
-200	25.12	4.9	85.95
	100.00	100.0	100.00

Fluidisation conditions were the same as for Table 2.

Calculations from the material balance indicated that the following amounts were removed during the run.

14.7% of - 65 + 100 mesh stone
24.4% " - 100 + 200 " "
86.6% " - 200 " "

Table 4

Batch Sizing Tests: 2.75-inch Reactor

Aspect Ratio 0.5:1 (Static Bed) Material Minus 10 mesh
Air Flow Rate 88.2 litres/min. Space Velocity 1.25 ft/sec.

Bed Charge				
Weight g	Weight Loss g	Weight Loss %	Pressure drop Inches W.G	Fluidisation Time Mins
155	-	-	1.8	0
111.5	43.5	28	1.4	10

Material Balance

Mesh Tyler	Feed% Cum	Size Stone % Cum	Dust Frac. % Cum
	100	72.0	28.0
10	1.48	3.8	
28	17.08	22.3	
35	32.52	42.6	
65	46.86	62.8	1.28
100	55.30	73.2	1.54
150	64.06	85.1	2.06
200	74.88	94.9	15.86
-200	25.12	5.1	84.14
	100.00	100.0	100.00

Dust losses were still continuing after 10 minutes of fluidisation.

Calculations from the material balance indicated that the following amounts were removed during the run.

11.4% of the - 65 + 100 mesh stone
20.1% " " - 100 + 200 " "
85.5% " " - 200 " "

Table 5

Batch Sizing Tests: 2.75-Inch Reactor

Aspect Ratio 0.5:1			Material - Minus 10 mesh	
Air Flow Rate 106 litres/min			Space Velocity-1.5 ft/sec.	
Bed Charge		Weight loss	Pressure drop	Fluidisation Time
Weight g	Weight loss g	%	Inches W.G	Mins
155	-	-	2.0	0
			1.55	5
			1.55	8
95.8	59.2	38.2	1.55	10

Material Balance

Mesh Tyler	Feed % Cum 100	Size Stone % Cum 61.8	Dust Frac. % Cum 38.2
10	1.48	4.8	
28	17.08	26.6	
35	32.52	44.6	
65	46.86	71.8)	0.2
100	55.30	83.0)	
150	64.06	92.8	5.4
200	74.88	96.4	25.0
-200	25.12	3.6	75.0
	100.00	100.0	100.0

The bed expansion was 4.6 inches when fluidised at the conclusion of the test. Fluidisation was violent during the first 5 minutes of dust loss.

Calculations from the material balance indicated that the following amounts were removed during the run.

35.2% of the - 65 + 100 mesh stone
67.6% " " - 100 + 200 " "
92.4% " " - 200 " "

Table 6

Batch Sizing Tests: 2.75-Inch Reactor

Aspect Ratio 0.5:1 (Static Bed)			Material - Minus 10 mesh	
Air Flow Rate 123.5 litres/min			Space Velocity 1.75 ft/sec	
Bed Charge		Weight	Pressure drop	Fluidisation Time
Weight g	Weight Loss g	%	Inches W.G	Mins
155	-	-	2.1	0
			1.75	2
			1.65	3
96.0	59.0	38.1	1.65	5

Material Balance

Mesh Tyler	Feed % Cum 100	Sized Stone % Cum 61.9	Dust Frac. % Cum 38.1
10	1.48	3.6	
28	17.08	23.6	
35	32.52	45.6	
65	46.86	68.4)	0.4
100	55.30	81.0)	
150	64.06	91.8	8.2
200	74.88	94.4	29.2
-200	25.12	5.6	70.8
	100.00	100.0	100.0

Violent fluidisation occurred during the period of dust loss, the dust losses being negligible after the first 3 minutes of fluidisation. The bed expansion at the conclusion of the test was 4.75 inches.

Calculations from the material balance indicated that the following amounts were removed during the run.

25.4% of the - 65 + 100 mesh stone
67.6% " " - 100 + 200 " "
88.3% " " - 200 " "

Table 7

Batch Sizing Tests: 2.75-Inch Reactor

Aspect Ratio 0.5:1 (Static Bed) Material - Minus 10 mesh
Air Flow Rate 141 litres/min. Space Velocity 2.0 ft/sec.

Bed Charge		Weight	Pressure drop	Fluidisation Time
Weight g	Weight Loss g	%	Inches W.G	Mins
155	-	-	2.25	0
			1.90	2
			1.85	4
89.0	66.0	42.6	1.85	5

Material Balance

Mesh Tyler	Feed % Cum	Sized Stone % Cum	Dust Frac. % Cum
	100	57.4	42.6
10	1.48	6.4	
28	17.08	35.2	
35	32.52	61.0	
65	46.86	81.4)	
100	55.30	89.6)	2.8
150	64.06	92.8	17.0
200	74.88	94.2	38.4
-200	25.12	5.8	61.6
	100.00	100.0	100.0

Violent fluidisation accompanied by heavy dust loss occurred in the first 2 minutes. At the end of the run fluidisation was normal giving a bed expansion of 5.0 inches.

Calculations from the material balance indicated that the following amounts were removed during the run

54.8% of the - 65 + 100 mesh stone
84.8% " " - 100 + 200 " "
88.8% " " - 200 " "

Table 8

Batch Sizing Tests: 2.75-Inch Reactor

Aspect Ratio 0.5:1 (Static Bed) Material - Minus 10 mesh
Air Flow Rate 123.5 litres/nin Space Velocity 1.75 ft/sec

Bed Charge		Weight Loss	Pressure drop	Fluidisation Time
Weight g	Weight Loss g	%	Inches W.G	Mins
155	-	-	2.05	0
			1.75	2
			1.65	4
			1.60	5
			1.55	10
90.7	64.3	41.5	1.55	15

Material Balance

Mesh Tyler	Feed % Cum	Sized Stone % Cum	Dust Frac. % Cum
	100	85.5	41.5
10	1.48	5.0	
28	17.08	26.4	
35	32.52	52.2	
65	46.86	74.4)	
100	55.30	87.0)	0.6
150	64.06	94.8	10.4
200	74.88	96.6	32.0
-200	25.12	3.4	68.0
	100.00	100.0	100.0

Fluidisation was violent during the first few minutes of high dust loss, returning to normal after a fluidisation time of 5 minutes and the bed expansion was 4.75 inches.

Calculations from the material balance indicated that the following amounts were removed during the run.

12.7% of the - 65 + 100 mesh stone
47.8% " " - 100 + 200 " "
91.6% " " - 200 " "

Table 9

Batch Sizing Tests: 2.75-Inch Reactor

Aspect Ratio 0.5:1 (Static Bed) Material - Minus 10 mesh
Air Flow Rate 123.5 litres/min Space Velocity 1.75 ft/sec

Bed Charge Weight g	Bed Charge Weight Loss g	Weight Loss %	Pressure drop Inches W.G	Fluidisation Time Mins
155	-	-	2.05	0
			1.90	2
			1.60	5
			1.55	10
			1.55	20
89.3	65.7	42.4	1.55	30

Material Balance

Mesh Tyler	Feed % Cum 100	Sized Stone % Cum 57.6	Dust Frac. % Cum 42.4
10	1.48	4.8	
28	17.08	28.0	
35	35.52	54.6	
65	46.86	78.0)	0.6
100	55.30	89.0)	
150	64.06	95.4	11.6
200	74.88	96.8	32.4
-200	25.12	3.2	67.6
	100.00	100.0	100.0

Fluidisation conditions were the same as for Table 8

Calculations from the material balance indicated the following amounts were removed during the run.

24.9% of the - 65 + 100 mesh stone
58.9% " " - 100 + 200 " "
92.7% " " - 200 " "

6.1.3 Continuous Fluidisation Tests on Two Screen Fractions

The minus 10 plus 65 mesh fraction initially fluidised well at 1.4 - 1.7 ft./sec space velocity with an aspect ratio of 0.5:1 (static). The initial bed charge was 128 gm. However, under continuous feed conditions trouble was experienced with feed tube blockages and bed sanding, so the air space velocity had to be gradually increased to a maximum of 2.14 ft/sec. after 20 minutes. The average pressure drop across the bed varied from 1.8 per cent. for ten minutes retention time to 3.7 per cent. for 60 minutes retention time as shown in Fig. 8.

The minus 10 plus 100 mesh fraction initially fluidised well at 1.4 - 1.7 ft/sec. space velocity, but again under continuous feed conditions, to avoid sanding and feed blackages the air space velocity had to be increased to 2.14 ft/sec. after 25-30 minutes fluidisation. The initial bed charge was 128 gm and the pressure drop across the bed varied from 2.65 - 2.85 inches W.G. Dust losses varied from 5 per cent. for 10 minutes retention time to 8.7 per cent. for 60 minutes retention time as shown in Figure 8.

6.1.4 Batch Calcination Tests

In the first calcination test using the minus 10 plus 80 mesh feed, the furnace took 15 minutes to reach 1000°C and on addition of the charge the temperature dropped to 650°C. A further seven minutes elapsed before the temperature recovered to 1000°C.

At this temperature the total pressure drop across the distributor plate and bed was 10.5" W.G. During the sampling period the average temperature was 1015°C.

However, approximately 48 hours elapsed between sampling and analysis, allowing reabsorption of H_2O and CO_2 from the air. Therefore this test was abandoned.

The total weight of lime produced from 170 g feed was 58.5 g.

In the second calcination test using the minus 10 plus 65 mesh feed, the temperature took 13 minutes to recover to 1000°C after addition of the feed. The average bed temperature during sampling was 1010°C and lime analyses are shown in Table 10.

Table 10

Lime Analyses: Minus 10 plus 65 mesh stone

Sample		Total CaO	CO_2 loss on ignition	Calculated	
Total time mins	Time after 1000°C mins	%		Available CaO	Unburned $CaCO_3$
12	2	96.6		96.6	
15	5	96.6		96.6	
20	10	96.3		96.3	
30	20	96.7	Nil	96.7	Nil
40	30	96.6		96.6	
50	40	96.5		96.5	
90	Dust sample	84.7	9.3	72.9	21.1
(Raw Limestone)		97.25 $CaCO_3$			

The total weight of lime produced from 170 g feed was 65.0 g. The minus 10 + 65 mesh feed for the calcination test represented 47 per cent. of the original feed approximately. Assuming complete calcination this represents a dust loss of 32 per cent. of the furnace feed or 15.0 per cent. of the original limestone feed for the minus 10 plus 65 mesh fraction.

∴ overall calculated dust loss including screening
= 53.0 + 15.0 = <u>68 per cent</u>.

This indicated that considerable attritioning and decrepitation of the particles occurred at high temperatures.

Hydration tests were carried out on the quicklime produced in both tests as follows:

(a) <u>Weight of hydrate formed</u>

Water was added to 20 g quicklime in a dish until all the lime appeared to be hydrated. Fifty per cent. excess water was added and the product dried for 3 hours at 110°C.

Weight of hydrate formed = 26.5 g

Percentage of original weight $= \frac{26.5 \times 100}{20} = 132.5\%$

Theoretically 100 parts of CaO should produce 133 parts of $Ca(OH)_2$.

(b) Rate of hydration

200 ml of water were added to an insulated beaker and 10 g of quicklime dropped into the water At the same time a stop clock was started and the solution constantly stirred with a mercury in glass thermometer. The temperature rise was noted at various time intervals, and the figures obtained were plotted in Figure 9.

The maximum temperature rise was 11.5°C in four minutes for the minus 10 plus 80 mesh cacine and 12.0°C in two minutes for the minus 10 plus 65 mesh calcine.

The quicklime produced was very white and appeared to be highly reactive when slaked in water. The dry hydrate appeared to be whiter than the original quicklime and had a very fine texture.

The calculated overall dust loss for the minus 10 + 65 mesh fraction related back to the original minus 10 mesh feed was approximately 68 per cent.

6.2 New Sample

6.2.1 Crushing Tests

Screen analyses of the crushed minus 10 mesh stone are shown in Table 11, and compared with those for the old sample used in previous tests.

Table 11

Sizing Analyses - New and Old Sample

Mesh B.S.S.	New Sample		Old Sample
	Fresh Mined % Cum	Aged 10 weeks % Cum	Aged 3 years % Cum
+ 10	0.75	0.20	1.48
14	4.65	4.60	
18	11.60	12.60	17.03
25	29.82	29.50	
36	55.57	52.15	32.52
52	72.52	69.25	
72	79.37	76.25	46.86
100	84.12	81.85	53.30
150	87.87	86.00	64.06
200	92.67	91.45	74.88
-200	7.33	8.55	25.12
	100.00	100.00	100.00

The crushing characteristics of the new sample were quite different from those of the old sample. The percentage minus 200 mesh material has been reduced and approximately 80 per cent. of the crushed product is plus 72 mesh.

The difference in sizing between fresh mined stone and stone age hardened for 10 weeks were negligible within the limit of reproducability.

The new sample produced only 7 - 8 per cent. minus 200 mesh material compared to 25 per cent. for the old sample

6.2.2 Fluidisation Characteristics: 2.75-inch Model

Results of the batch fluidisation tests carried out on the minus 10 mesh material are shown in Fig. 10.

Space velocities, in the range of 1.7 - 2.0 ft/sec. were required to fluidise this material due to its porosity. Dust losses occurred simultaneously with channelling in both cases.

6.2.3 Continuous Fluid Bed Sizing Tests

Results of the sizing tests are shown in Tables 12, 13 and 14 respectively. The sizing tests indicated that a suitable air-sized feed can be produced for the calcination stage using an air space velocity of 2.5 - 3.0 ft./sec. The dust losses were of the order of 15-20 per cent. giving a fraction containing 96 - 98 per cent. plus 100 mesh, of which approximately 81 per cent. was minus 18 plus 72 mesh material.

Table 12

Sizing Tests: 2.75-Inch Reactor

Av. Feed Rate 13.8 g/min: Space Velocity 2.1 ft/sec:
Av. Retention time 9.3 mins: Air flowrate 147 litres/min:
Aspect Ratio 1:1 Fluidised: Av. Δ P = 2.25" W.G.

Mesh B.S.S.	Feed % Cum. 100	Sized Stone % Cum 85.4	Dust % Cum 14.6
+ 10	0.75	0.25	
14	4.65	2.15	
18	11.60	7.20	
25	29.32	23.90	
36	55.57	53.75	
52	72.52	76.25	
72	79.37	85.80	1.6
100	84.12	93.10	2.8
150	87.17	97.50	10.0
200	92.67	98.95	46.6
-200	7.35	1.05	53.4
	100.00	100.00	100.0

Calculations from the material balance figures show that the minus 72 plus 100 mesh fraction has been increased by 31 per cent: the minus 100 plus 200 mesh fraction reduced by 41.5 per cent. and the minus 200 mesh fraction reduced by 87.8 per cent.

Table 13

Sizing Tests: 2.75-Inch Reactor

Av. Feed Rate 14.2 g/min: Space Velocity 2.5 ft/sec
Av. Retention time 9.1 min: Air flowrate 175 litres/min.
Aspect Ratio 1:1 fluidised: Av. Δ P = 2.65" W.G.

Mesh B.S.S.	Feed % Cum 100	Sized Stone % Cum 84.5	Dust % Cum 15.5
+ 10	0.75	0.57	
14	4.65	3.65	
18	11.60	11.57	
25	29.82	33.47	
36	55.57	65.42	
52	72.52	85.77	
72	79.32	91.66	0.16
100	84.12	96.77	4.52
150	87.87	98.54	19.05
200	92.67	99.11	51.35
-200	7.35	.89	48.65
	100.00	100.00	100.00

Calculations from the material balance figures show that the minus 72 plus 100 mesh fraction has been reduced by 10.3 per cent: the minus 100 plus 200 mesh fraction reduced by 75.8 per cent; and the minus 200 mesh fraction reduced by 89.8 per cent.

Table 14

Sizing Tests: 2.75-Inch Reactor

Av. Feed Rate 15.0 g/min: Space Velocity 3.0 ft./sec.
Av. Retention time 8.6 min: Air flowrate 210 litres/min.
Aspect Ratio 1:1 fluidised: Av. ΔP = 3.0" W. G.

Mesh B.S.S.	Feed % Cum 100	Sized Stone % Cum 79.5	Dust % Cum 20.5
+ 10	0.75	0.30	
14	4.65	3.05	
18	11.60	10.28	
25	29.82	29.83	
36	55.57	60.23	
52	72.52	83.98	0.15
72	79.32	92.68	1.05
100	84.12	97.68	11.95
150	87.87	98.48	29.45
200	42.67	98.90	60.60
-200	7.35	1.10	39.40
	100.00	100.00	100.00

Calculations from the material balance figures indicate that the minus 72 plus 100 mesh fraction has been increased by 5 per cent; the minus 100 plus 200 mesh fraction reduced by 85.8 per cent. and the minus 200 mesh fraction reduced by 85.0 per cent.

6.2.4 Batch Calcination of Air-sized Minus 10 mesh Fraction

With the stone in the furnace from the beginning of the test, the furnace took 23 minutes to heat up to 1000°C. The average bed temperature during sampling was 1010°C. Lime analyses together with the head sample analysis are given in Table 15.

Table 15

Lime Analyses - Air Sized Minus 10 Mesh Stone

Sample		Total CaO	CO_2 loss on Ignition	Calculated	
Total time in furnace min.	Time after 1000° C min	%	%	Unburned $CaCO_3$	Available CaO
23	Zero	97.5	0.55	1.25	96.8
28	5	97.7	0.38	0.86	97.2
33	10	98.0	0.22	0.50	97.7
40	Dust*	86.4	13.60	30.9	69.1

Raw Limestone Feed 98.0% $CaCO_3$; 1.37% $MgCO_3$; 0.2% SiO_2; and 0.4% Al_2O_3 + Fe_2O_3

*Dust sample obtained from top ring of furnace.

The total weight of lime produced from 170 g feed including samples was 81.5 g.

The air sized minus 10 mesh feed for this calcination test represented 84.5 per cent. of the original feed material.

Assuming complete calcination and no dust loss the theoretical lime weight should be 95.4 g.

∴ Percentage dust loss during calcination

$$= \frac{(95.4 - 81.5)}{95.5} \times 100 = \underline{14.6 \text{ per cent.}}$$

∴ Calculated overall dust loss including the air-sizing step

$$= 15.5 + \frac{(84.5 \times 14.6)}{100} = 15.5 + 12.35 = 27.85 \text{ per cent.}$$

say <u>28 per cent</u>.

This dust loss was less than half that obtained with the old sample in the previous tests.

A rate of hydration test on this lime gave a temperature rise of 12°C in 30 seconds (See Fig. 9). However the calcine did not break down to a sludge on slaking, as it retained its original particle shape and size. These slaked particles were quite resistant to crushing between the fingers, indicating that over-burning may have taken place.

6.2.5 Multistage Fluidisation: 2.75-Inch Reactor

Attempts to obtain continuous stage to stage transfer of solids in this reactor were unsuccessful. Although good fluidisation was obtained in each of the three stages, continuous flow down the transfer tubes was not obtained.

Due to the low specific gravity of the stone and high porosity, the air passing through the transfer tubes kept the stone particles in "teeter" thus stopping the flow by gravity to the next stage.

On increasing the air flow beyond 3 ft/sec. solids were often sucked from the second stage up the transfer tube and into No.1 stage.

Variation of the feed tube height above the distributor plate in each stage had little effect.

6.2.6 Multistage Fluidisation: 6-Inch Reactor

Pressure drop tests carried out on the two types of air distributor plates indicated that at the air flow rates required for good stone fluidisation, the pressure drop across the smaller diameter nozzles would be too great for efficient operation. For a 320 litres/minute air flow-rate the pressure drop across the smaller nozzles was 56 inches W.G. as against 2.0 inches W.G. for the larger nozzles. The plates containing the large diameter nozzles were used for the fluidisation test work.

Pressure drop vs air flow-rate figures obtained from the batch fluidisation of the minus 10 plus 100 mesh stone are shown plotted in Figure 11. Good fluidisation was obtained at 320 - 375 litres/minute with little slugging and dust losses due to attrition appeared to be low.

It will be noted that the above air flow-rate corresponded to a space velocity of 0.97 - 1.12 ft./sec. which is considerably less than the space velocity of 2.0 ft./sec. required in the 2.75-inch reactor.

Attempts to obtain continuous stage to stage solids transfer when fluidising three stages were again unsuccessful in this equipment due to fluidisation or "teetering" of the stone particles in the transfer tubes.

With the air flow-rate set at 325 litres/minute good fluidisation was obtained in each of the three stages. Immediately continuous feed was started, stone began to flow down the transfer tube between Nos. 1 and 2 stages until a slug of stone approximately 4 - 6 inches long built up in the tube. This slug of stone then became fluidised and was blown back into the top stage carrying some stone from the lower bed. Unless the reactor was bumped by hand the transfer tube would then remain empty with air short circuiting through it, even when a solids head of several inches had built up above the transfer tube in No.1 stage.

Variation in size of the transfer tubes and height of the feed point above the distributor plates had no effect upon this phenomenon.

6.2.7 Fluidisation Tests in Cross-Flow Reactor

Results of the batch fluidisation using a 3-inch bed depth of minus 10 plus 100 mesh stone are shown plotted in Figure 12. Good fluidisation was obtained at an air flow rate of 180 litres/minute, representing a space velocity of 1.26 ft./sec. The total pressure drop through the bed at the above air flow-rate was 5.5 inches W.G. of which 2.8 inches W.G. was due to the plate.

Dust losses appeared to be high at air flow-rates over 160 litres/minute. When fluidised the bed expansion was 5 inches.

In the continuous tests, attempts to obtain continuous solids transfer from cell to cell with countercurrent gas flow failed. Within a few minutes of commencing feed, the solids in Nos. 2, 3 and 4 cells were blown back through the transfer ports into No.1 cell until it was completely filled.

The explanation for this phenomenon is that the pressure drop across each solids transfer port, was less than the sum of the pressure drops across each preceding distributor plate, stone bed and air transfer tube respectively.

Attempts were then made to increase the pressure drop through the transfer ports by closing the baffles to the smallest opening possible for solids transfer. This opening was found to be 1/16 inch. For an air flow rate of 180 litres/minute through each cell the pressure drop across each transfer p ort would have to be greater than 5.5 inches W.G. (across plate and stone bed) plus the pressure drop in the air transfer tube.

At the above air flow-rate, measured pressure drops across baffle openings of 3/16" x 1½" and 1/16" x 1½" were found to be 1.25 inches W.G. and 4.3 inches W.G. respectively. At openings of less than 1/16" it was impossible to pass the coarser solids. These results indicated that true countercurrent conditions could not be obtained in this type of reactor.

However, continuous cross-flow feed of solids could be obtained when using parallel air flow to each cell. The total air flow required in the reactor for fluidisation was 630 - 640 litres/minute. With the baffle openings set at 3/16", the maximum feed rate obtained through the reactor was 115 g/minute, corresponding to a retention time of approximately 7 minutes per cell.

The measured pressure drops and bed expansion figures for each cell are listed below.

Cell No.	Pressure Drop Inches W.G.	Bed Expansion inches
1 (feed end)	5.0	4½
2	4.1	4
3	3.7	3¾
4 (discharged end)	5.8	3½

The higher pressure drop in No.4 cell was due to build-up of coarse material necessitating a higher gas flow for fluidisation.

The gradual decrease in pressure drop for the other three cells reflected the differential head of fluidised solids necessary to maintain flow of solids.

7. DISCUSSION AND RECOMMENDATIONS

The experimental work carried out on the old limestone sample has shown that although a high grade lime can be produced in a batch fluidised bed calciner, the dust losses involved during the crushing, air sizing and calcination steps were excessive. These losses were of the order of 65 - 70 per cent. of the original minus 10 mesh feed. The overall dust losses in a typical fluid-bed plant in the United States would not exceed 25 per cent.

The optimum feed size for fluidisation was found to be minus 10 plus 65 mesh Tyler.

Fluid bed air sizing of the minus 10 mesh material produced a fraction containing 81.4 per cent. plus 65 mesh and 89.6 per cent. plus 100 mesh stone, representing only 56.4 per cent. of the original feed.

During the calcination the lime particles became soft and this led to considerable attrition losses in the bed at high temperatures. These losses were of the order of 32 per cent. of the furnace feed or 15.0 per cent. of the original minus 10 mesh feed.

The quicklime produced from this old sample was highly reactive when slaked with water, producing a fine textured hydrated lime. The lime before hydration rapidly absorbed water and carbon dioxide from the atmosphere if left undovered.

The new sample although similar in grade to the old was coarser grained and during crushing to minus 10 mesh broke up readily to its natural particle size.

The production of fines was appreciably less than that for the old sample, approximately 80 per cent. being coarser than 72 mesh B.S.S. as against 47 per cent. for the latter.

Age hardening this stone over a period of ten weeks had a negligible effect on the crushing characteristics. However, if aged for a period of twelve months or more the difference could be quite large giving rise to a higher production of fines.

Fluidisation of the new sample required higher air flow-rates than for the old sample due to its high porosity and coarse particle size. A satisfactory air-sized feed fraction could be prepared for calcination, by batch fluidisation at an air space velocity of 2.5 - 3.0 ft./sec. and a retention time of approximately 9-10 minutes.

The dust losses produced from air sizing were still high, being 15-20 per cent. of the feed fraction. This sized material of which 81 per cent. was minus 18 plus 72 mesh B.S.S. fluidised well in the 2.75-inch reactor at 140 litres/min or 2.0 ft./sec. space velocity.

Although lime could be produced containing over 97 per cent. available CaO, considerable attritioning of the lime particles occurred during calcination. These dust losses consisted of a mixture of unburned calcium carbonate and burnt lime containing less than 69 per cent. available CaO, and amounted to 14.6 per cent. of the furnace feed. The overall dust loss from air sizing and batch calcination was approximately 28 per cent. for a total fluidisation time of 50 minutes. This figure is not excessive when compared with that for the old sample and the 25 per cent. losses experienced in some fluid-bed plants in the United States.

However, the results of continuous multistage fluidisation in both the 2.75 and 6-inch reactors were negative in that stage to stage transfer of the solids was not obtained due to fluidisation of the particles in the transfer tubes.

This phenomenon was due to the fact that the material was of low density and porous so that high air flow-rates were needed for fluidisation. Sufficient head of stone particles could not be maintained in the transfer tubes to balance the pressure drop across the two beds and in most cases it would just "teeter" in the tube or be blown up into the top stage.

Lower air space velocities were required to fluidise the new sample in the 6-inch equipment than for the 2.75-inch equipment. The difference was of the order of 0.9 ft./sec. The reason for this is thought to be due to increased wall friction effects in the smaller reactor.

Attempts to obtain continuous stage to stage transfer of solids under countercurrent gas flow in the cross flow type fluid bed reactor were unsuccessful, due to pressure drop troubles explained previously. However, with parallel air flow to each cell, stage transfer of solids can be obtained, making this type of reactor suitable for drying and air sizing operations only.

In conclusion it is considered that calcination of Mt. Gambier limestone by the usual fluidisation technique would not be practicable. It is suggested that since this limestone is very soft in nature, some method of calcination in the fine state, such as flash calcination in cyclones, should be investigated as a further phase in the programme.

The Department of Industrial Development in Western Australia has carried out a few preliminary laboratory scale flash calcination tests on aeolian type limestone sands with a moderate degree of success.

APPENDIX

Alternative Methods of Calcination for Mount Gambier Polyzoal Limestone

1. CONVENTIONAL SHAFT KILN PROCESS

This method would not be suitable due to the low crushing strength of sized lumps of Mt. Gambier stone. There would also be considerable wastage of small stone in the feed separation step.

However, Oamaru Lime Ltd., in New Zealand have a small pilot plant operating at the moment on a similar type of limestone, employing the use of a specially designed low shaft kiln. The process suggested by Dr. N.V.S. Knibbs in the U.K. consists of fine grinding and pelletisation of the ground stone with a suitable binder and coal dust. The resulting pellets were said to be sufficiently strong to be burned in a special vertical kiln.

The economics of pelletisation would have to be investigated closely before employing this method.

2. ROTARY KILN PROCESS

This method usually requires closely sized feed of not less than 1/4" size and would involve high dust losses in the crushing and screening operations.

In the kiln itself, due to the soft nature of the stone, dust losses due to attritioning of the stone particles caused by the rotary action of the kiln would be high.

This process would therefore be unsuitable for this type of stone unless fine grinding and pelletisation of the feed could be used as mentioned above.

Rotary kilns have been used throughout the world for the re-burning of lime sludges produced from the sulphate pulp and sugar beet industries with considerable success. However in thest cases the wet sludge contains dissolved alkali salts which tend to act as binding agents for the lime sludge in the drying section of the kiln. The sludge is apparently quite resistant to break-down in the calcination zone and consequently dust losses are low.

3. MULTIPLE HEARTH FURNACE PROCESS

Multiple hearth furnaces of the Herreschoff and Skinner type have been used for calcination of some limestones, including lime sludges from the sulphate pulp and sugar beet industries.

However the capacity is lower than conventional kilns and fuel consumption higher.

High maintenance costs are also associated with this type of furnace.

It is thought that in the case of the polyzoal limestone, considerable break-down of the particles would occur by the rabbling action on the hearths. Dust losses would then occur during solids transfer from hearth to hearth.

The use of multiple hearth type furnaces is therefore not recommended for Mt. Gambier limestone calcination.

4. CALCIMATIC FURNACE PROCESS

This type of furnace has a circular horizontal hearth and was developed by the Calcimatic Co. of Toronto, Canada for the calcination of soft friable limestones.

In this process the stone, preferably of 1/4" minimum sizing, is fed evenly on to a revolving circular hearth where it remains in the stationary state until after calcination. A fixed refractory hood is mounted over the hearth and is sealed by means of a water or sand trap on either side of the hearth. This hood is divided into several compartments by means of baffles for preheating, calcination and cooling of the stone.

Air for combustion is fed into the combustion chamber countercurrent to the burnt lime movement. The combustion gases then flow back through the stone preheating compartment and eventually are taken up through the stone feed and preheat bin.

The final cooled lime is removed from the hearth by means of a cross scraper conveyor.

The estimated heat requirement for this type of furnace is approximately 8 million B.T.U's per ton of lime, thus comparing favorably with conventional rotary kilns. The addition of heat recovery equipment reduced this figure to 6 million B.T.U's per ton.

Finely divided limestones are usually pelletised before calcination in this type of furnace.

This furnace can produce a high available CaO product and can produce lime with predetermined chemical and physical properties, and is infinitely variable with respect to (a) stone size (larger than 1/4"), (b) stone quality, (c) fuel type and (d) production rate.

This type of process would therefore be suitable for the calcination of Mt. Gambier polyzoal limestone.

5. MODIFIED TYPE "Dwight-LLoyd" SINTERING MACHINE PROCESS

This process was investigated by the U.S. Bureau of Mines in 1926-27 using modified 12" and 24" wide pallet machines. Both solid fuel firing with coal dust and liquid fuel firing with kerosene were used, employing downdraught flow of combustion gases through the stone bed.

Due to chilling of the stone in immediate contact with the grate bars, a dressing of previously burnt lime was used. The stone charge was graded with the finest size on the bottom and the coarser sizes on the top to achieve even calcination rates.

To minimise over-burning of the lime on the surface, fixed ploughs were placed in two rows of four across the windbox, such that the charge was continually turned over or rabbled.

Although high grade lime of over 90 per cent. available CaO was produced from these machines, the total fuel consumption was high being of the order of 10-11 million B.T.U's per ton of lime, and troubles were experienced with fluxing of the coal ash with the lime.

The machine was found to have quite a high capacity with up to 300 lb. CaO/square foot grate area/24 hours. Considerable losses of minus 20 mesh stone occurred through the grate bar into the windboxes, thus requiring specially designed grate bars for stone finer than 20 mesh.

It is probable that the polyzoal limestone could be calcined successfully on such a machine provided fine grinding and pelletisation of the feed was carried out.

Maintenance and fuel costs would be higher than for the Calcimatic process which would also be more efficient and flexible.

6. FLASH CALCINATION IN CYCLONES

This type of process presents an entirely new approach to calcination and it is not known at this stage whether any commercial plant employing flash calcination exists in the world today.

However the Department of Industrial Development in Western Australia, has carried out a few preliminary laboratory scale flash calcination tests on "aeolian" type limestones with some degree of success.

The process envisaged would consist of a series of cyclones arranged for countercurrent flow of combustion gases and solids. The actual flash calcination would occur in a tall vertical tube furnace, oil fired, which would deliver the flash calcined product into the cyclones for gas-solids separation.

The solids discharge from each cyclone would be controlled at the bottom by means of air lock feeders, after which they would be picked up by the gas stream from a preceding cyclone and blown into another cyclone for further heat recovery and gas-solids separation. Any number of cyclone stages could be used to give a good overall heat recovery.

The feed sizing used in such a process would probably be finer than 52 mesh thus enabling the entire crushed stone feed to be used for calcination.

With the stone in such a fine state seconds only should be required for complete calcination.

Oil firing could be used in the furnace.

It is recommended that any further work on the calcination of polyzoal limestones should proceed along the above lines.

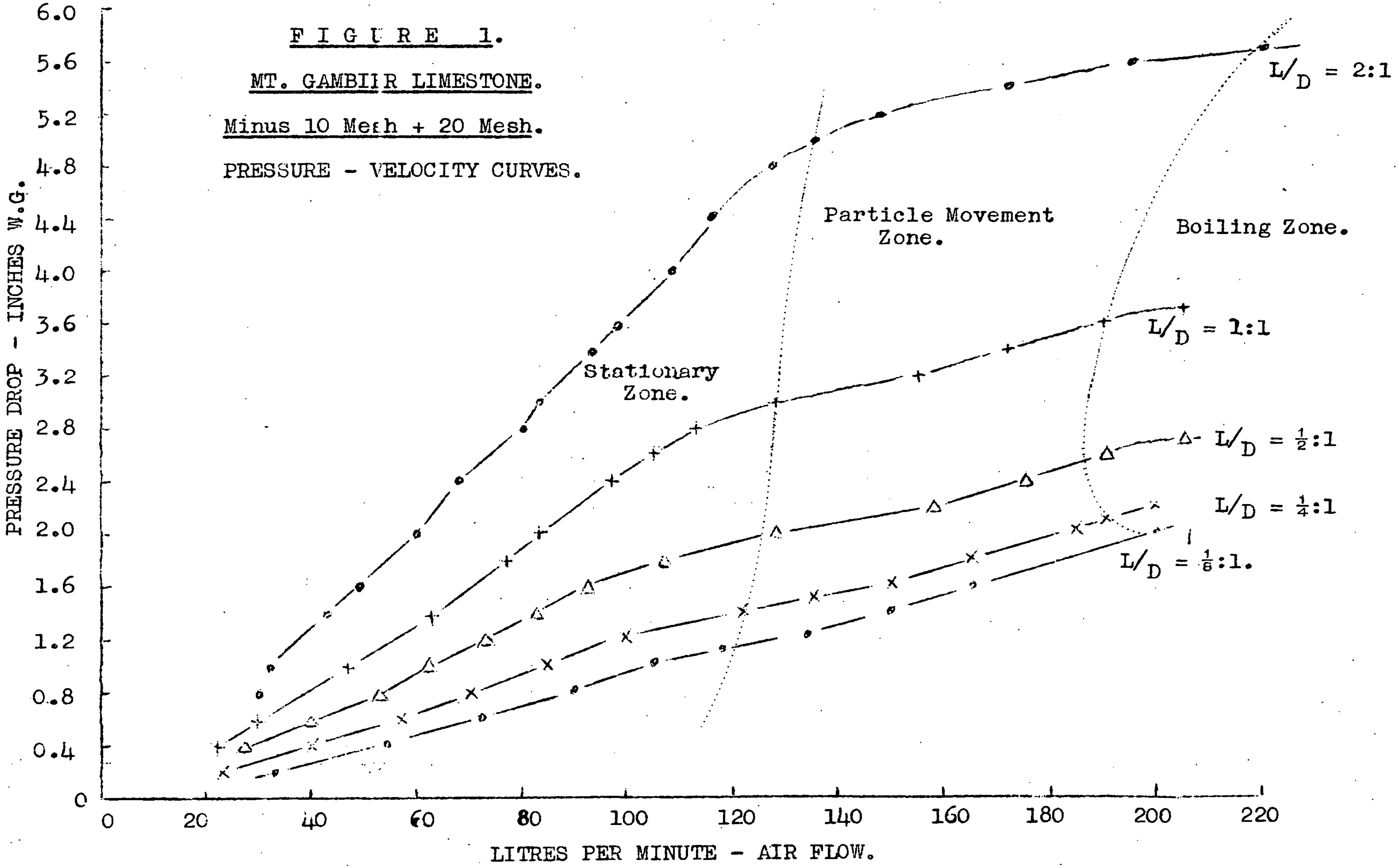


FIGURE 1.

MT. GAMBIER LIMESTONE.

Minus 10 Mesh + 20 Mesh.

PRESSURE - VELOCITY CURVES.

FIGURE 2.

MT. GAMBIER LIMESTONE.

Minus 20 Mesh plus 48 Mesh Tyler.

PRESSURE - VELOCITY CURVES.

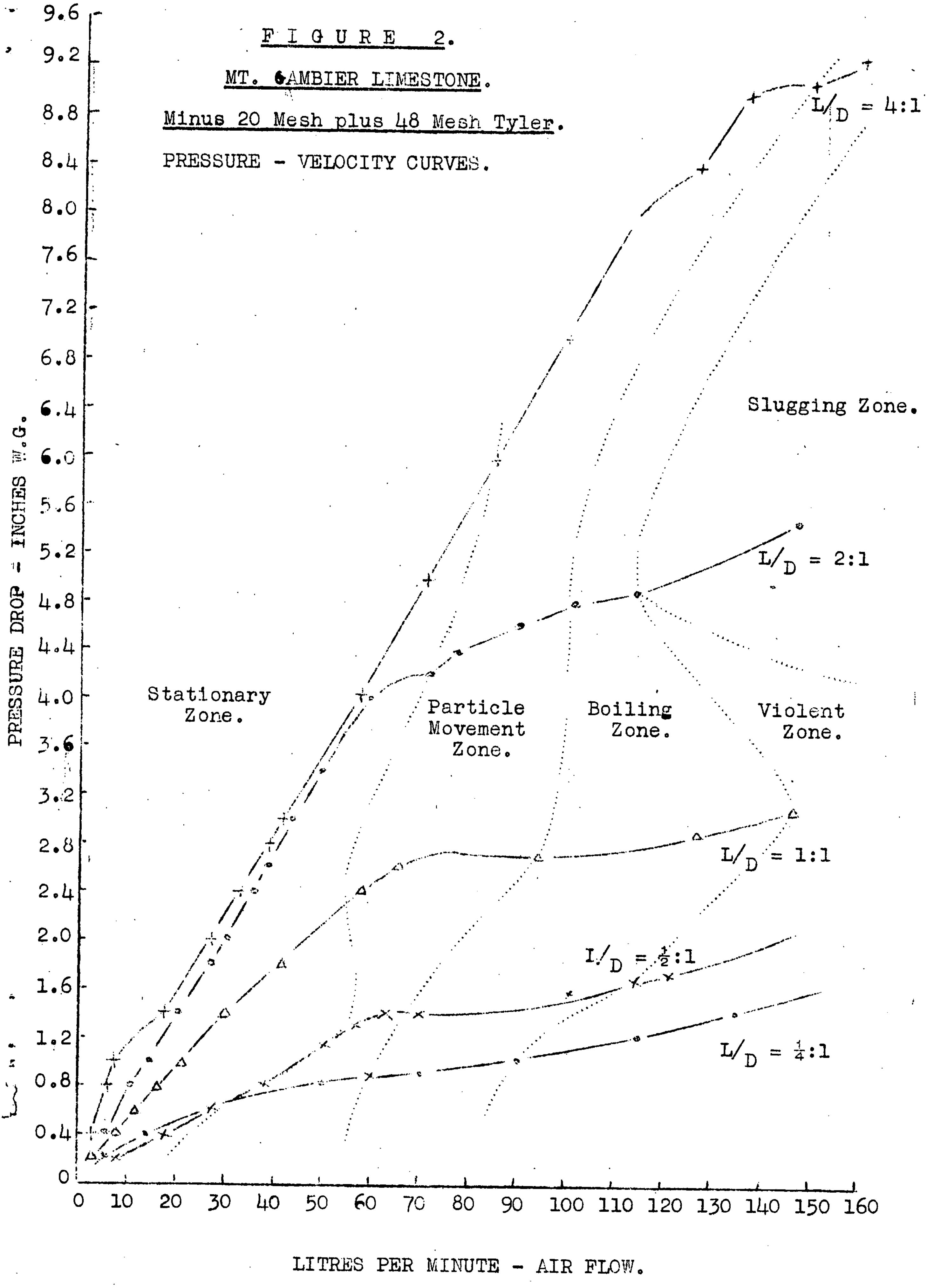


FIGURE 3.

MT. GAMBIER LIMESTONE.

Minus 48 Mesh plus 55 Mesh.

PRESSURE - VELOCITY CURVES.

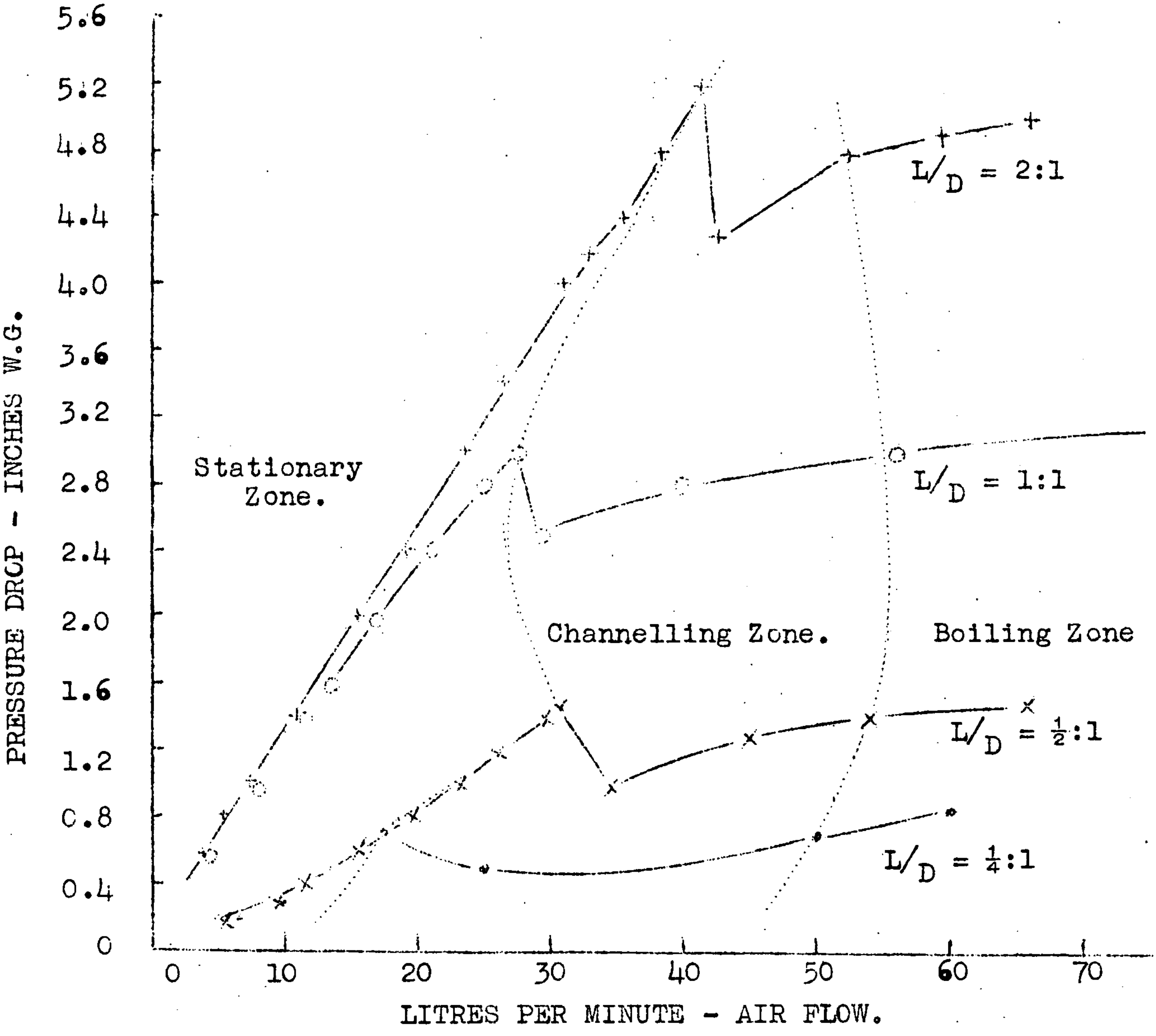
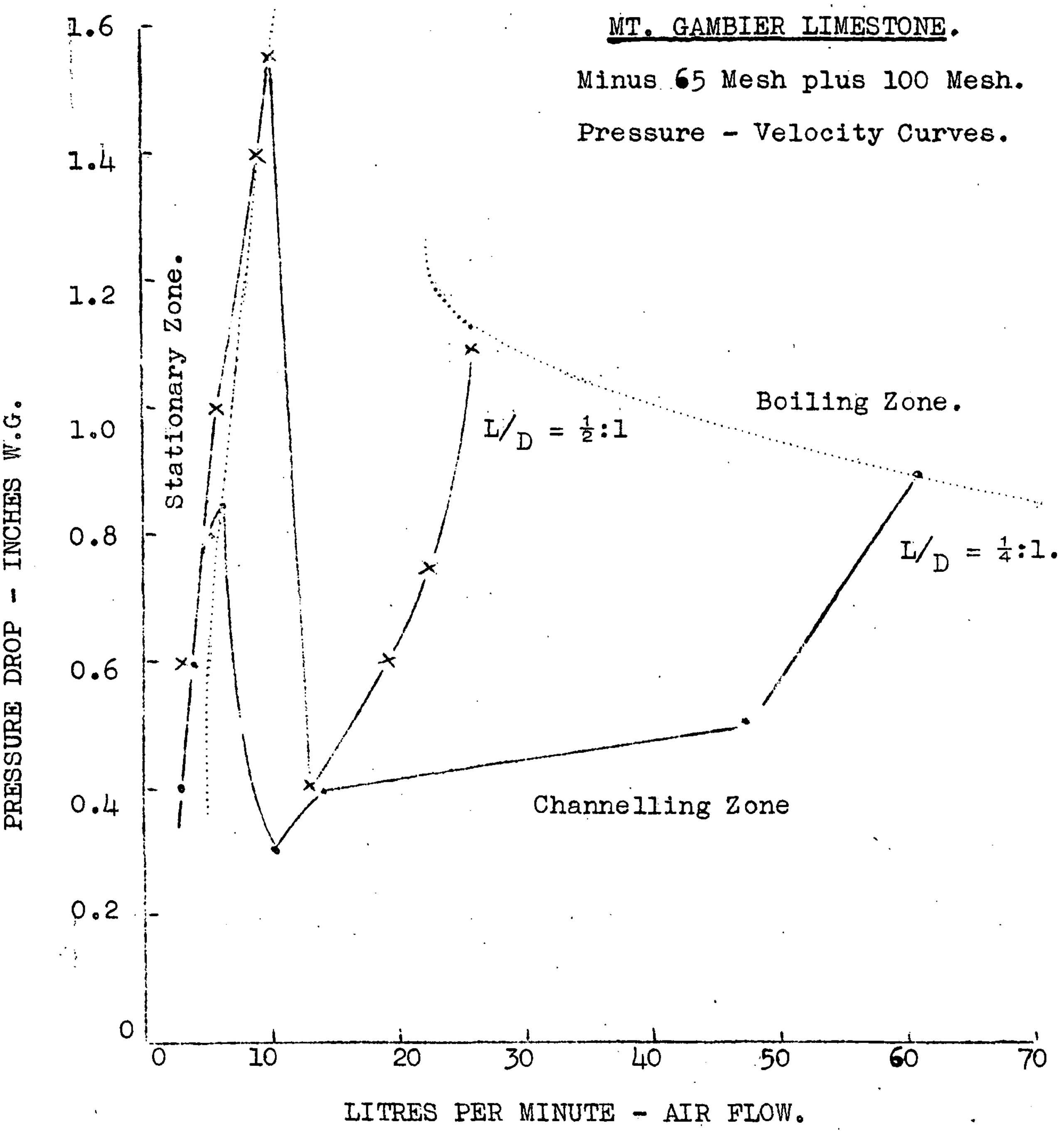


FIGURE 4.

MT. GAMBIER LIMESTONE.

Minus 65 Mesh plus 100 Mesh.

Pressure - Velocity Curves.



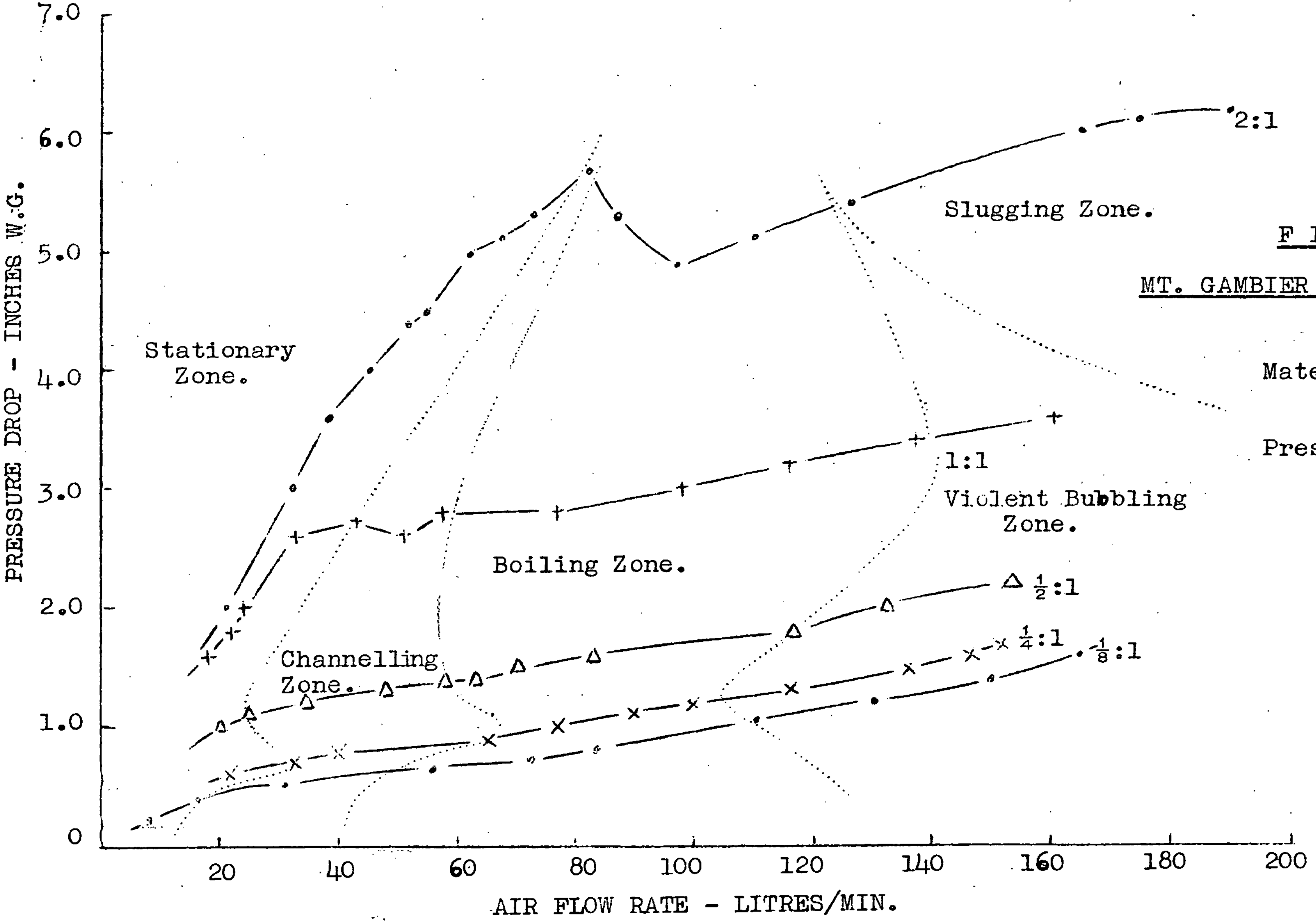


FIGURE 5.

MT. GAMBIER LIMESTONE FLUIDIZATION TESTS.

Material: Minus 10 Mesh
+ 65 Mesh Tyler.

Pressure - Velocity Curves.

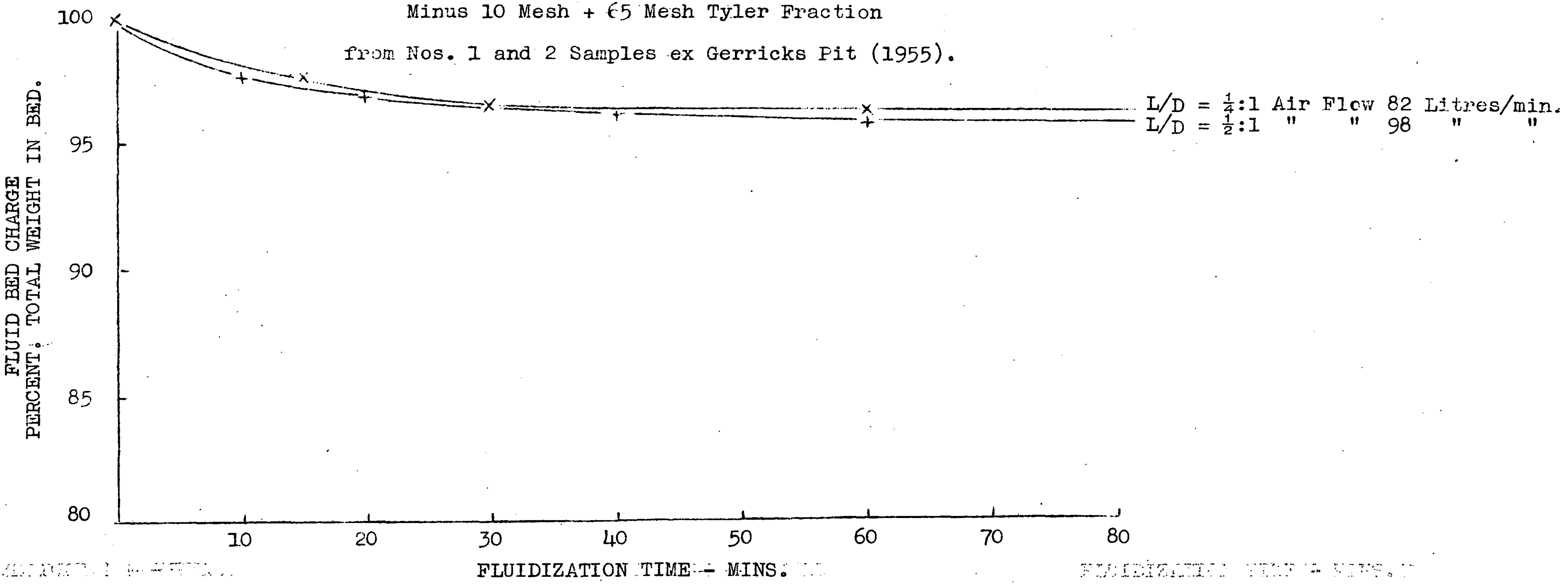
FIGURE 6.

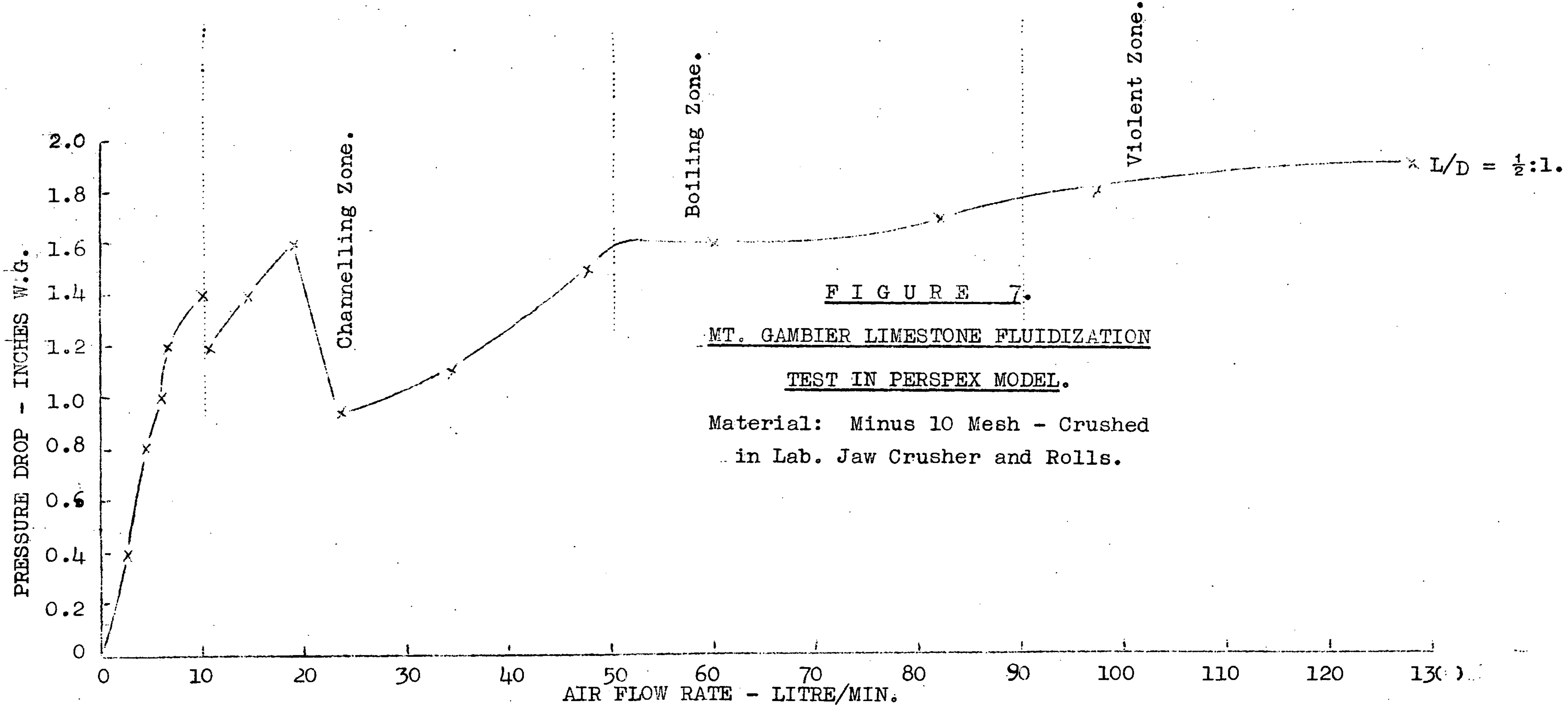
MT. GAMBIER LIMESTONE -

DUST LOSSES DUE TO FLUIDIZATION.

Minus 10 Mesh + 65 Mesh Tyler Fraction

from Nos. 1 and 2 Samples ex Gerricks Pit (1955).





F I G U R E 7.

MT. GAMBIER LIMESTONE FLUIDIZATION TEST IN PERSPEX MODEL.

Material: Minus 10 Mesh - Crushed in Lab. Jaw Crusher and Rolls.

PRESSURE VELOCITY CURVE.

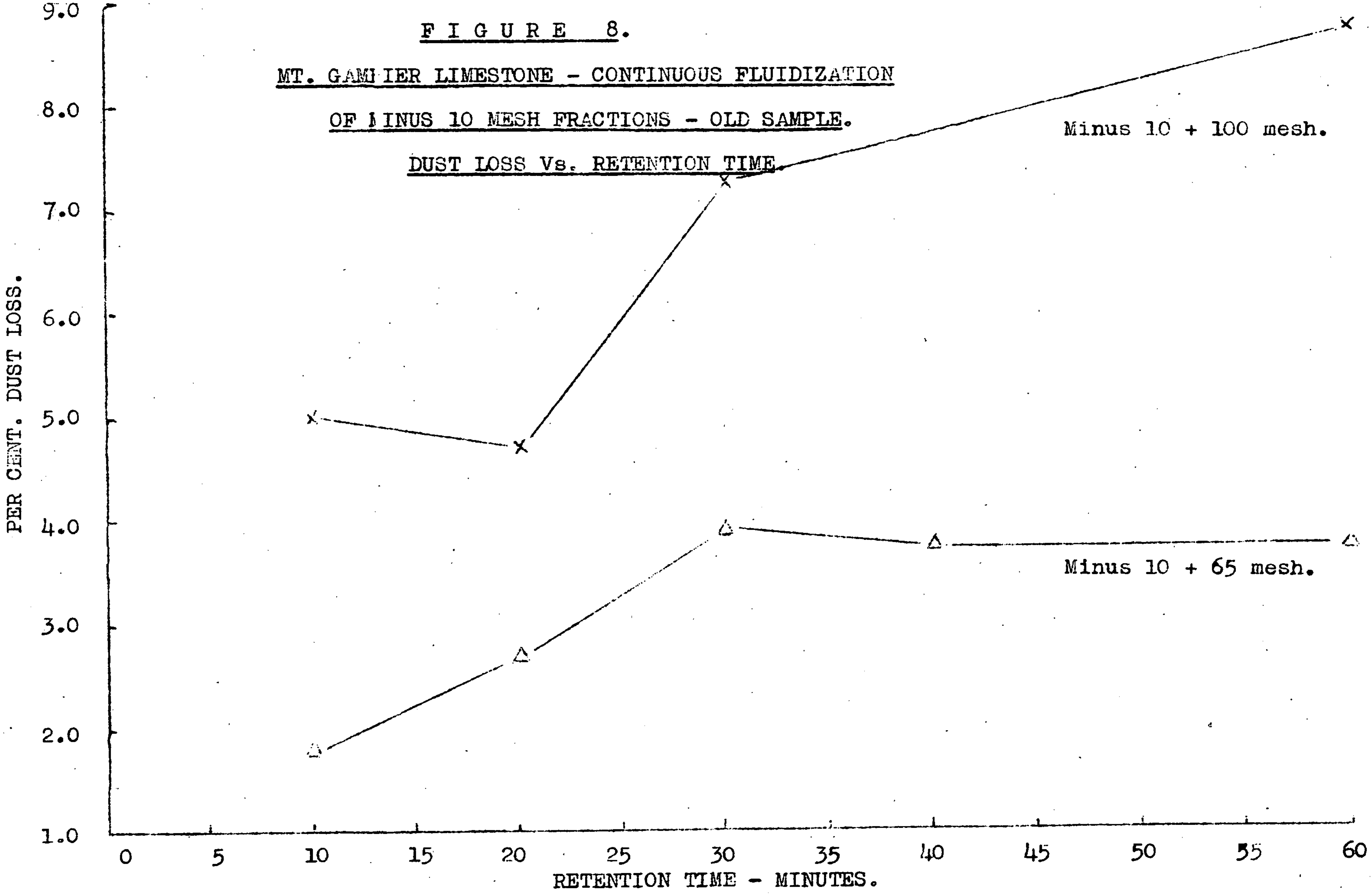


FIGURE 8.

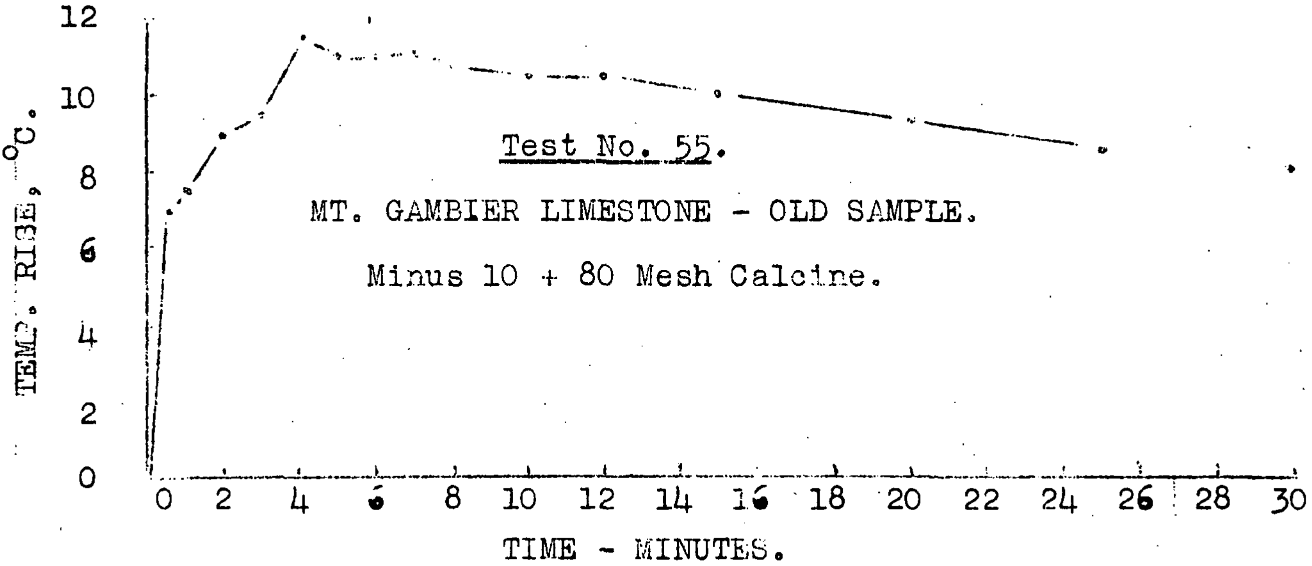
MT. GAMBIER LIMESTONE - CONTINUOUS FLUIDIZATION OF MINUS 10 MESH FRACTIONS - OLD SAMPLE.

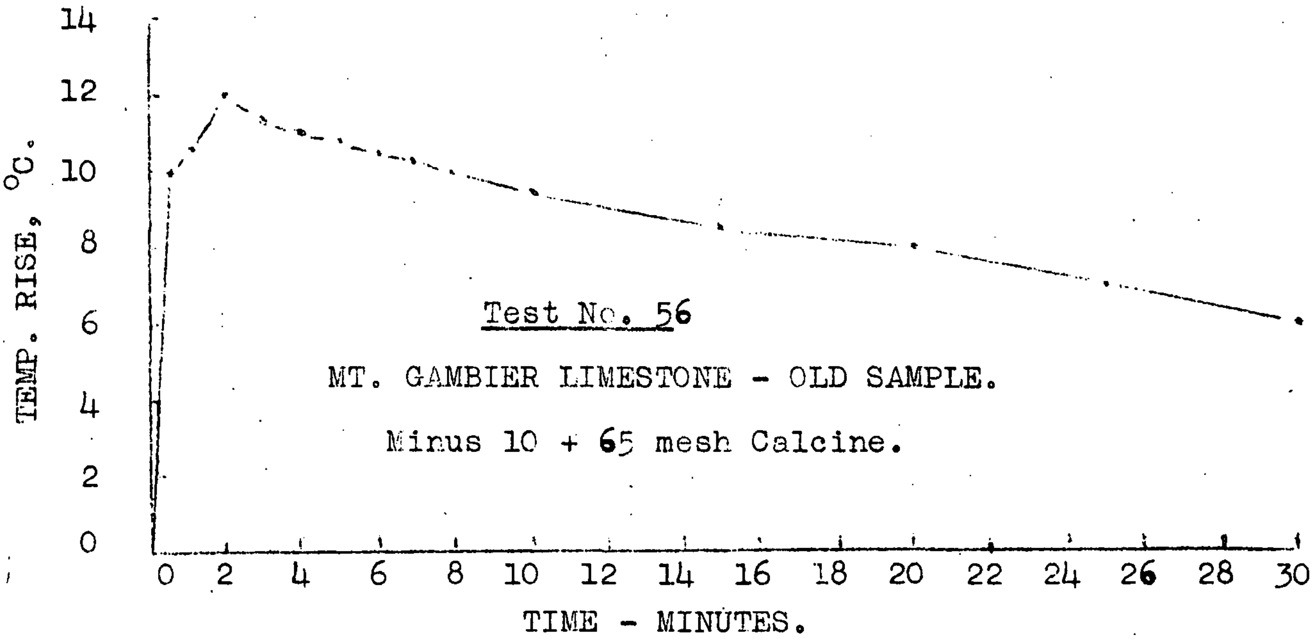
DUST LOSS Vs. RETENTION TIME.

FIGURE 9.

MT. GAMBIER LIMESTONE - CALCINES.

RATE OF HYDRATION CURVES.





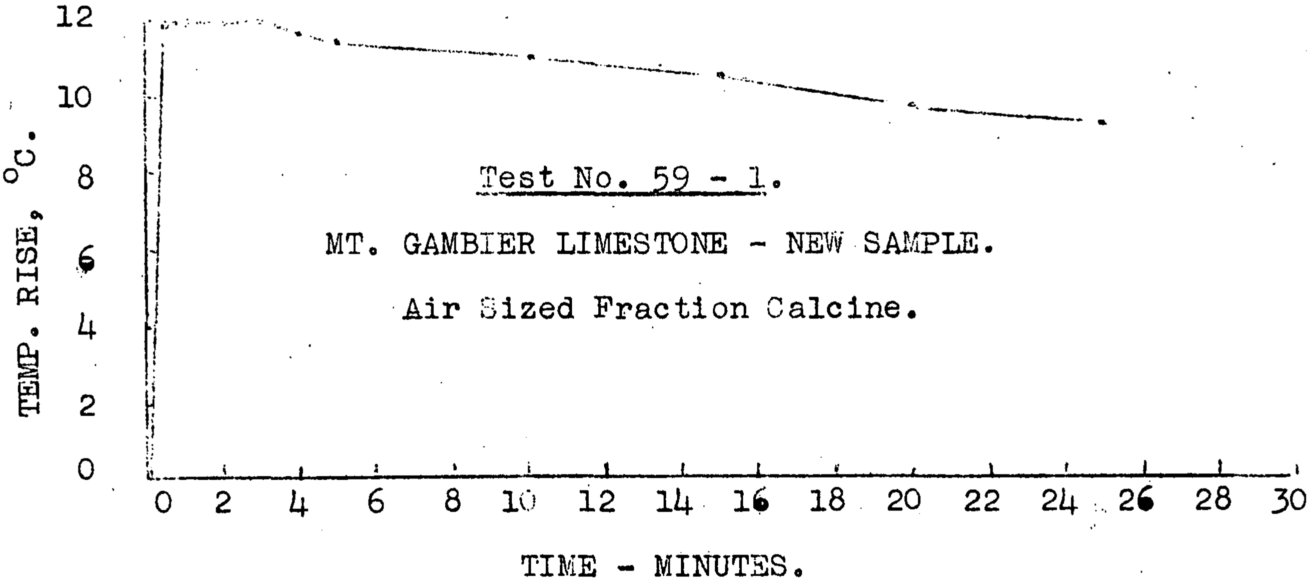
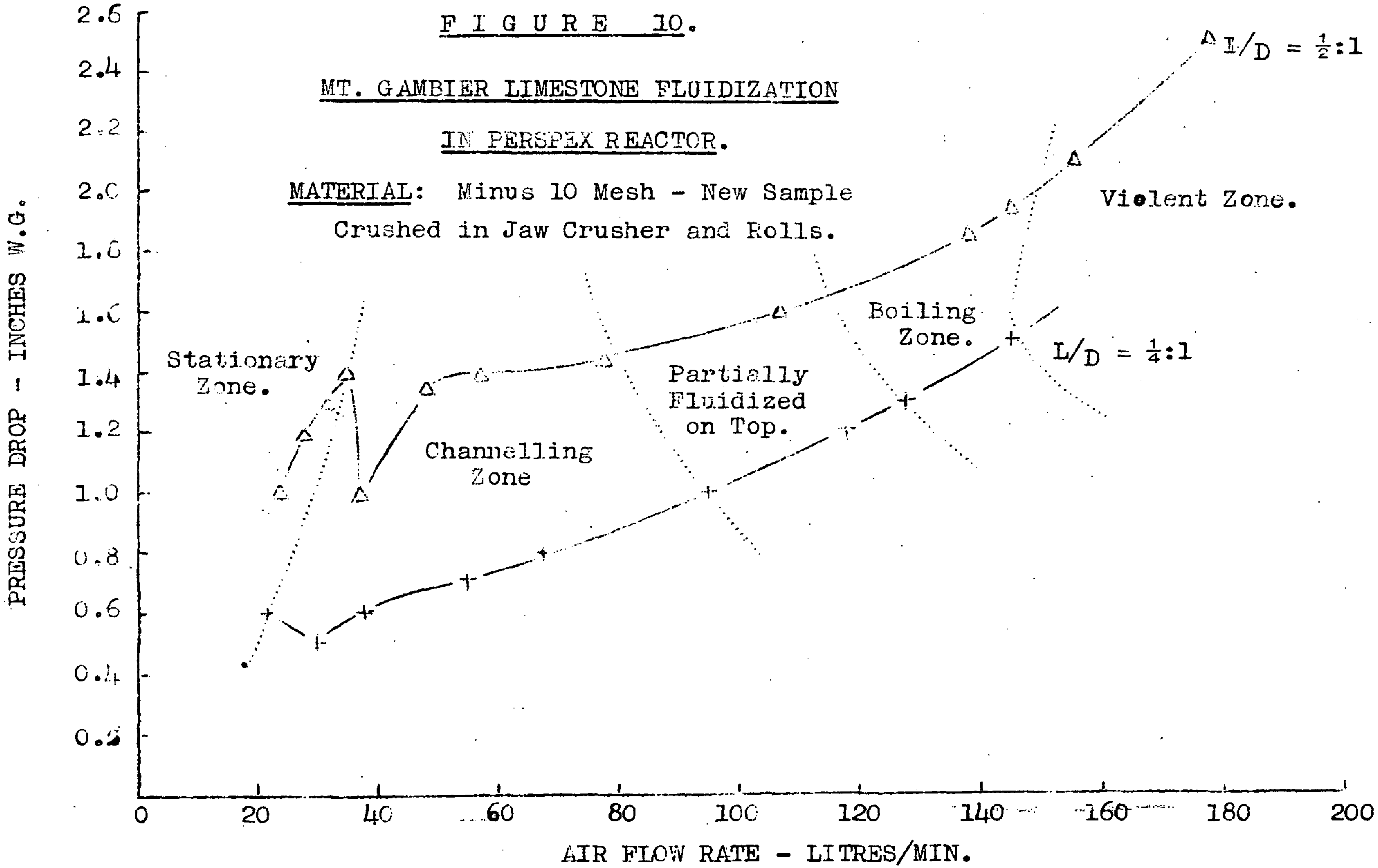


FIGURE 10.

MT. GAMBIER LIMESTONE FLUIDIZATION IN PERSPEX REACTOR.

MATERIAL: Minus 10 Mesh - New Sample
Crushed in Jaw Crusher and Rolls.



PRESSURE-VELOCITY CURVES.

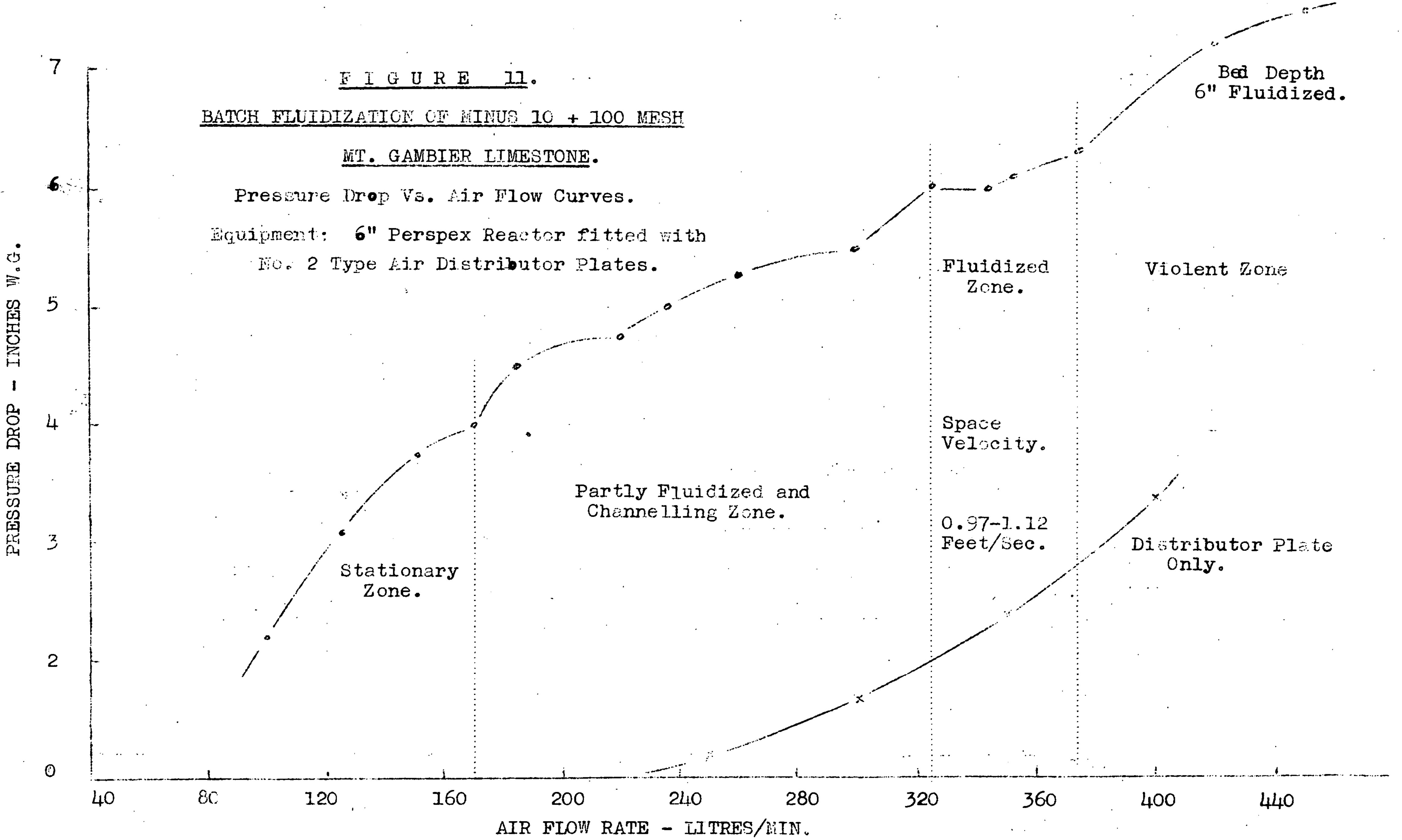


FIGURE 11.

BATCH FLUIDIZATION OF MINUS 10 + 100 MESH

MT. GAMBIER LIMESTONE.

Pressure Drop Vs. Air Flow Curves.

Equipment: 6" Perspex Reactor fitted with
No. 2 Type Air Distributor Plates.

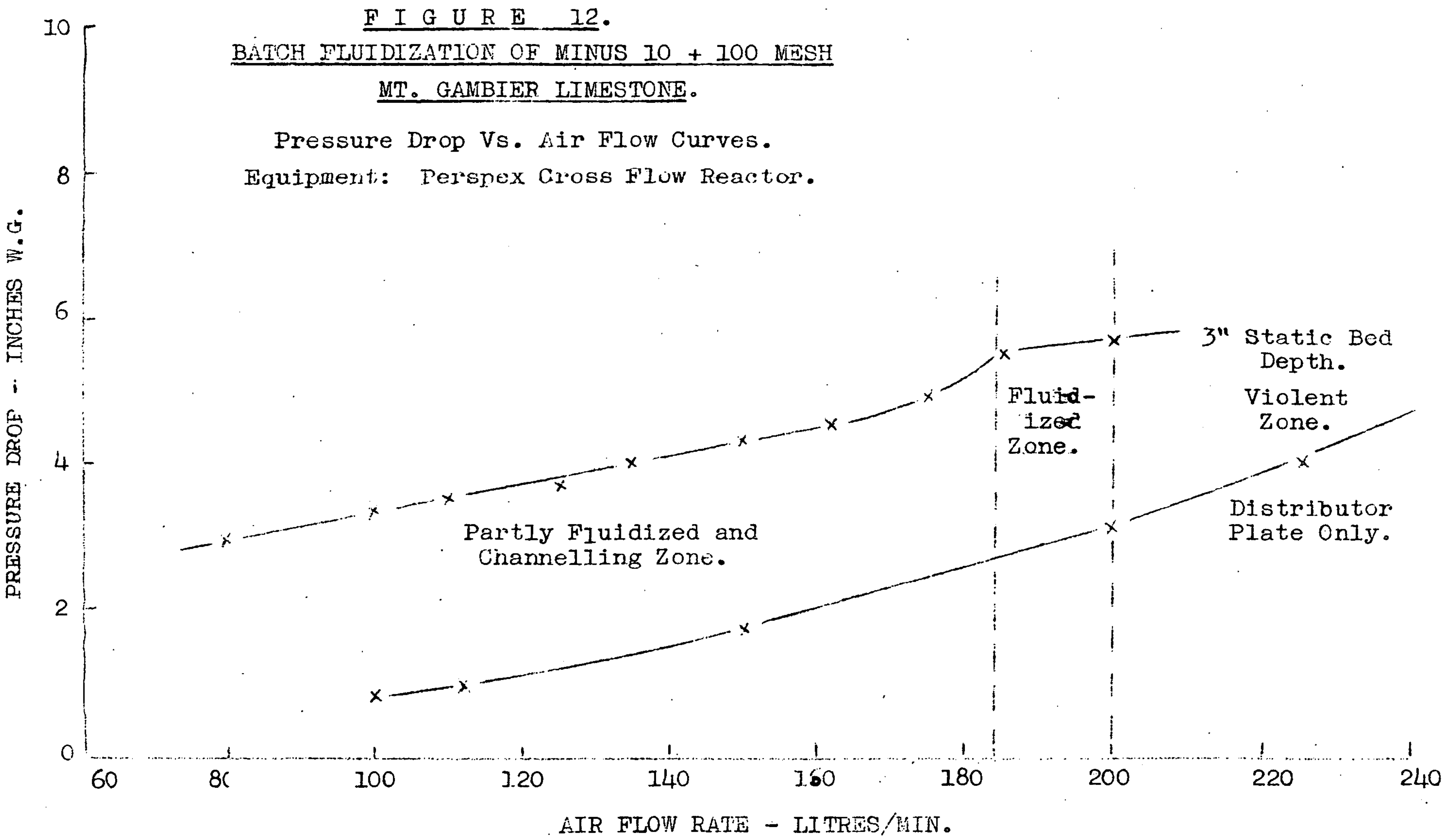
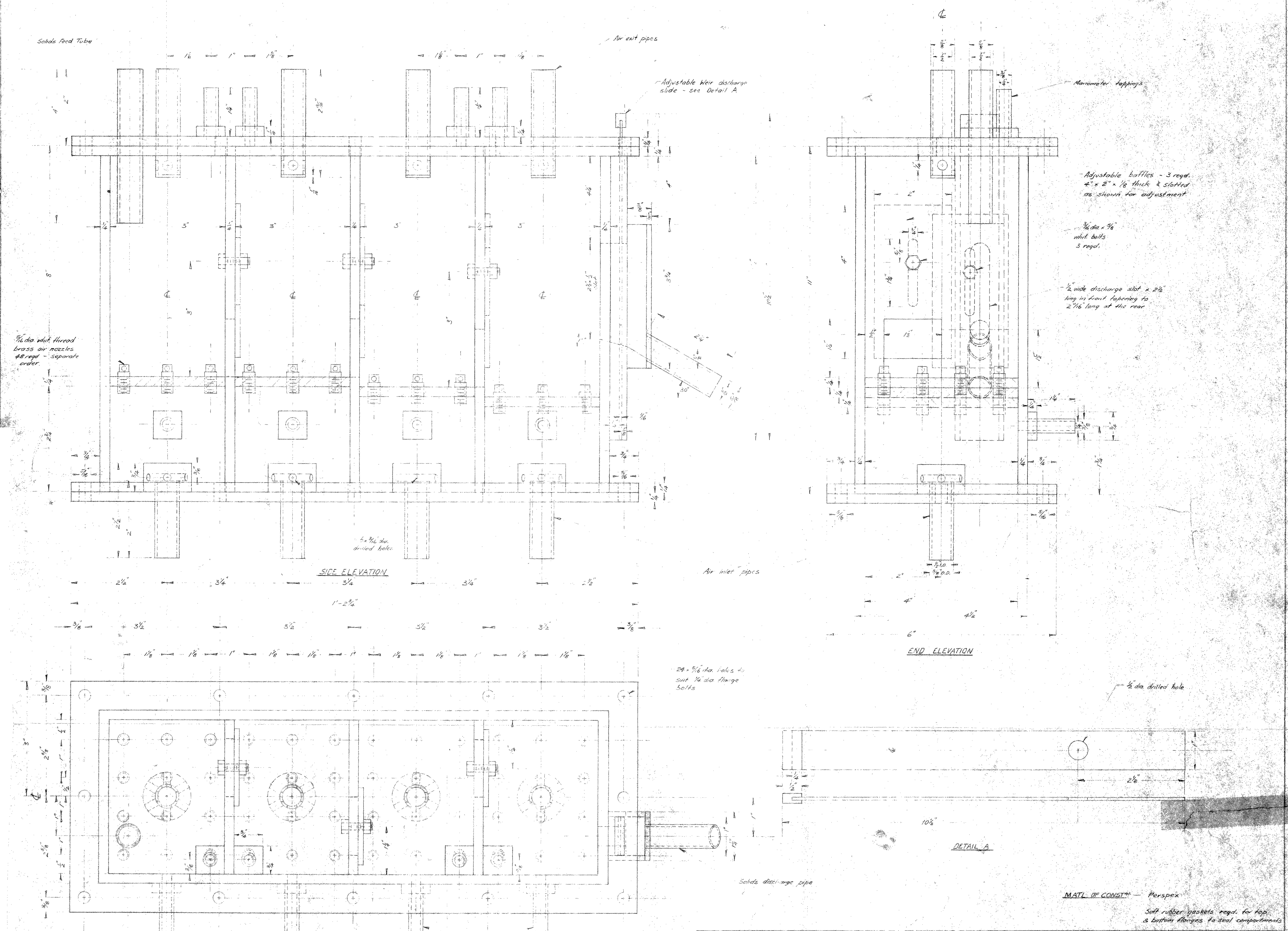


FIGURE 12.

BATCH FLUIDIZATION OF MINUS 10 + 100 MESH MT. GAMBIER LIMESTONE.

Pressure Drop Vs. Air Flow Curves.

Equipment: Perspex Cross Flow Reactor.



Manometer tappings

PLAN VIEW

AUST. MINERAL DEV. LABS.	Approved	Passed		Scale - Full Size
EXPERIMENTAL CROSS FLOW FLUID BED REACTOR			Drn. J.McE.H Tcd. Ckd. Exd.	RC-349 Date: 10th July 1959