

Hydrodynamic studies in solids-liquid contacting

by

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ABSTRACT.

A new technique for countercurrent contacting of fluidized solids and liquid has been studied. Liquid flow through a cascade of stages gives fluidized or spouted beds of solids. This flow is periodically reversed to convey solids in dense flow between adjacent stages. Conditions under which solids hold-up in the stages of a cascade would be stable have been determined by studying reverse flow with a single stage. The effects on solids transfer between stages of reverse flow rate, volume of reverse flow, and of allowing solids to settle before reverse flow have been investigated. Silver sand and ion-exchange resin have been used as solids.

The vertically upward dense phase flow of -36+52 B.S.S. mesh silver sand and ballotini in $\frac{1}{2}$ inch and $\frac{3}{4}$ inch pipes has been investigated. It has been shown that, at solids velocities of less than about 2 feet/sec., the pattern of pressure drop of conventional solids conveying with up to 35 percent by volume solids is followed. That is, the absolute pressure drop less that due to static head of solids and liquid is the same as the frictional pressure drop of pure liquid flowing at the mean velocity of solids and liquid. At solids velocities of greater than 2 feet/sec., a solids - wall frictional mechanism

has been shown to account for linearly increasing pressure drop with increasing solids velocity. The velocity of the slip water for any combination of solids and pipe diameter has been shown to be a constant fraction of the solids velocity at all but the lowest solids velocities. The difference in slip velocities between the two pipe sizes has been accounted for by the difference in the opposing forces applied to the solids.

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Nomenclature.

Dense phase flow - flow of solids and liquid which is initially vertically downward out of a vessel and where the solids fraction is not less than in the free-settled state.

Dilute phase flow - flow of solids and liquid where the solids fraction is less than that of free-settled.

Units. C.g.s. units have been used wherever possible in Part I though where f.p.s. units applied to manufacturers dimensions, etc., these have been retained. In Part II, f.p.s. units have been used to be consistent with the existing data on dilute phase flow. One exception to this has been in the calculation of solids fraction in the X-ray attenuation density gauge where the conventional c.g.s. units have been used.

Symbols for Part I.

- A - volume of the retainer plus transfer lines (litre)
- c - constant element of volume (litre)
- e_d - water fraction by volume of dense phase flow delivered from the single stage.
- e_t - voidage or water fraction by volume in the retainer.
- F - forward flow rate (litre/min.)
- H - free-settled volume of solids initially present in the single stage (litre)

- H_{crit} - the smallest value of H to give V_{max} (litre)
- m - operating gradient, the average slope of the almost linear section of the sloping part of the V versus H plot
- Q - size of a reverse flow volume (litre)
- Q_s - $Q - A$ (litre)
- R - reverse flow rate (litre/min.)
- S - slip ratio, i.e. The absolute velocity of slip water relative to solids as a percentage of solids velocity.
- T - settling time between forward and reverse flow (sec.)
- U - fraction of retainer volume filled with free-settled solids at the end of reverse flow with an initial hold-up of solids of H_{crit}
- V - the volume of free-settled solids transferred out of the single stage in one reverse flow period (litre)
- V_{max} - maximum free-settled volume of solids which can be transferred out of a stage in one reverse flow period when all variables other than solids hold-up are constant (litre)

Symbols for Part II.

- A_w - relative attenuation of γ -rays by water in test section compared to the attenuation with a vacuum in the test section.

- A_x - cross-sectional area of the test line (ft.²)
 B - number of right angled bends in the test line
 C - total count of γ -photons in one counting operation
 C_D - drag coefficient on an individual solids particle
 (dimensionless)
 D - pipe diameter (ft.)
 DP - measured pressure drop between PI_1 and PI_2 (fig. 31)
 (cm. Hg/H₂O)
 d_p - particle diameter (ft.)
 e_d - water fraction by volume of delivered dense phase flow
 e_{dil} - water fraction by volume of delivered dilute phase flow
 e_t - voidage or water fraction by volume in the test line
 F_d - drag force on an individual solids particle
 (lb. ft./sec.²)
 f_b - free-settled solids fraction by volume of ballotini
 h - height (ft.)
 I_{ov} - paralysis corrected count rate of γ -photons with a
 vacuum in the test section (count/sec.)
 I_{sw} - paralysis corrected count rate of γ -photons with
 dense phase flow in the test section (count/sec.)
 I_w - paralysis corrected count rate of γ -photons with water
 in the test section (count/sec.)
 L_1 - change in liquid level in metering cylinder (cm.)
 L_s - change in solids level in metering cylinder (cm.)

- N - number of solids particles
- ODP - measured pressure at PI_3 (fig. 31) (lb./in.² gauge)
- ~~ODP~~_t - measured pressure at PI_3 corrected for water static head (lb./in.² gauge)
- P3 - measured pressure at PI_1 (fig. 31) (lb./in.² gauge)
- P3_t - measured pressure at PI_1 corrected for water static head (lb./in.² gauge)
- P_s - solids pressure exerted by dense phase flow on the pipe walls
- Re/ - particle Reynolds number
- S - slip ratio, i.e. The absolute velocity of slip water relative to solids as a percentage of solids velocity
- S_c - solids static pressure between PI_1 and PI_2 (fig. 31) (cm. Hg/H₂O)
- t - duration of a dense phase flow run (sec.)
- t_p - paralysis time of counting equipment (μsec.)
- V_{dil} - dilute phase mean velocity of solids and liquid (ft./sec.)
- V_p - free-settling velocity of solids particles (ft./sec.)
- V_s - mean absolute velocity of solids particles (ft./sec.)
- V_{slip} - mean absolute velocity of liquid past solids particles (ft./sec.)
- W_{den} - work done in lifting solids in dense phase

- W_{dil} - work done in lifting solids in dilute phase
 x_{pl} - path length in the density gauge test section (cm.)
 x_s - "depth" of solids in the density gauge test section (cm.)
 x_w - "depth" of water in the density gauge test section (cm.)
 ΔP_d - dynamic pressure drop. i.e. The measured pressure drop over the vertically rising section of the test line less the static head of liquid and solids (ft. water head/ft. pipe length)
 ΔP_{dil} - water frictional pressure drop in dilute phase flow (ft. water head/ft. pipe length)
 ΔP_t - total pressure drop. i.e. The measured pressure drop over the vertically rising section of the test line less the static head of liquid (ft. water head/ft. pipe length - except in deriving drag coefficient when lb./ft. sec.² is used)
 μ - coefficient of sliding friction between dense phase solids and the pipe walls
 μ_b - mass absorption coefficient for Co-60 γ -rays attenuated by ballastini (cm.²/gm.)
 μ_s - mass absorption coefficient for Co-60 γ -rays attenuated by solids (cm.²/gm.)

- μ_w - mass absorption coefficient for Co-60 γ -rays
 attenuated by water ($\text{cm.}^2/\text{gm.}$)
 ρ_b - density of ballotini (gm./ml.)
 ρ_{Hg} - density of mercury (gm./ml.)
 ρ_l - density of liquid (~~gm./ml.~~)
 ρ_s - density of solids (gm./ml.)
 ρ_w - density of water (gm./ml.)

Symbols for statistical analyses.

- d - divisor of total square effect to give mean square effect
 F - the standard variance ratio
 N - number of readings pooled
 S - the statistic
 S' - statistic transformed for analysis
 ν_1 - degrees of freedom of an effect
 ν_2 - degrees of freedom of a residual
 $\bar{X}$ - signifies a multiple of the average of the statistics at all levels of X with other independent variables constant.
 X_m^i - signifies an m^{th} order effect of X
 $X_m^i Y_n^j$ - signifies an interaction of the m^{th} order effect of X with the n^{th} order effect of Y .

$X_m Y_n Z_o$ - signifies the m^{th} level of factor X, the n^{th} level of factor Y, and the o^{th} level of factor Z. It indicates the setting of independent variables in an experimental run.

PART I.

HYDRODYNAMIC STUDIES OF A NEW TECHNIQUE OF
SOLIDS - LIQUID CONTACTING.

1. INTRODUCTION.

1.1. Origin of the technique.

The concept studied is a technique for the continuous treatment of particulate solids with a countercurrent liquid stream using a series of fluidized beds. It was devised by Cloete and Streat (1,2,3), originally to deal with the problems associated with highly radioactive solutions.

Initially, the concept was also considered for solids-gas contacting. Solids-liquid contacting was selected for investigation since, firstly, this was a simpler system being free from significant changes in the fluid volume with pressure drop across the unit. In itself, the variation of volume with pressure makes it seem unlikely that the technique would be widely applicable to solids-gas contacting. Secondly, settled solids were known to be difficult to move in a solids-gas system and to give rise to large pulsating pressure drops (4).

1.2. Developments in solids-liquid contacting.

1.2.1. Fixed beds.

Conventionally, solids-liquid contacting has been carried out with fixed beds of particulate solids through which the liquid stream flows downwards. This is essentially a discontinuous technique with solids being progressively treated and with most of the solids-liquid exchange occurring in a fairly narrow band in the bed. A degree of exhaustion of the unused solids capacity is always reached beyond which further exchange becomes increasingly difficult and the liquid output consequently deteriorates. In conditions of unfavourable equilibrium of the exchange process, deterioration begins soon after the start of passing liquid through an untreated column. Similarly, the latter part of regeneration of the solids in a fixed bed is wasteful of reagents. However, fixed bed treatment is completely satisfactory for many processes where large quantities of liquid have to be passed through the bed to exhaust it and interruptions are relatively rare. Fixed beds are also satisfactory if the reacting materials are cheap and a continuous liquid output of constant composition is not essential.

Continuity of output and fairly constant composition can be achieved with groups of identical fixed

beds. The various liquid streams are rotated between beds and the same liquid streams may be passed through two or more beds in different states of exhaustion of exchange capacity. This multiple grouping of fixed beds is widely used in industry and is most satisfactory for many processes. However, the solids inventory is many times larger than the quantity of solids involved in exchange with liquid streams at any one time. This becomes a significant consideration with high cost materials such as ion-exchange resins. Understandably, the development of synthetic ion-exchange resins after the Second World War has led to many designs of plant to give more truly continuous solids-liquid contacting.

1.2.2. Moving beds.

The most widely developed type of continuous solids-liquid contactors are those based on moving packed beds of solids with counter-current liquid flow. It is usual in those processes where solids are continuously cycled to use two moving beds (5) but, unlike the fixed bed technique, several different solids treatment operations can be carried out in a single moving bed. (6). A well-designed moving bed would have a much higher proportion of the solids inventory engaged in mass transfer

at any time than would occur with the corresponding fixed bed.

The simplest arrangement of moving packed beds are descending solids beds moving under gravity and the many variations of this technique are primarily concerned with methods of solids withdrawal from the base of the bed. Interest in this technique dates back to the work of Matthiessen in 1885 (7). A rotating disc (7), a valve for handling and metering solids (8), a screw (9), and various methods of water ejection (5, 10, 11, 12) have been used to effect solids withdrawal. But, simple descending beds are very limited in liquid throughput rates since the liquid stream must not fluidize the packed bed. Fluidization rapidly mixes the solids and destroys the countercurrent nature of the technique. This difficulty may be overcome in two ways. Either the solids may be restrained at the top of the packed bed or an ascending solids bed may be used. Porter (10) follows the former approach and uses a screen at the top of the solids bed. Liquid flow is regularly stopped to move solids downwards. The Sun Oil Company (13) are the patentees of a novel technique of ramming solids downwards against a countercurrent liquid stream by surmounting this processing section with a section in which there is a high

velocity liquid stream co-current with solids movement.

The alternative approach of ascending solids beds is typified by the Higgins contactor (6,14) and that of Ericksen and Pike (15,16). In the Higgins contactor, the downward liquid flow is periodically stopped and the solids bed hydraulically jerked upwards. The technique of Ericksen and Pike uses alternating movement of solids upwards and liquid downwards at between 5 and 15 cycles per minute and although rather simpler than the Higgins contactor would probably be less suitable for simultaneous processing of several liquid streams in different sections of the same column of solids.

In general, it would be desirable with moving bed techniques to avoid any mechanically moving parts such as valves and screws in contact with packed solids since hard solids would cause wear and friable materials would tend to be broken. Friability is an important consideration for ion-exchange resins and favours the use of techniques where the solids are moved hydraulically (5, 10, 13, 16). The Higgins contactor has been compared with fixed bed techniques using ion-exchange resins and is claimed to be economically attractive where high purity or high recovery is required (17).

All techniques using packed beds - both fixed and moving - suffer to some degree from reduced solids-

liquid contact due to channelling in the bed. This is aggravated if fines are present in the liquid stream and permanent fixed beds must be periodically backwashed (fluidized) to free them from accumulated fines. Where the fines are particularly prolific and difficult and expensive to remove by filtration, even moving packed beds may be unsuitable and other techniques have been devised to deal with these circumstances.

1.2.3. Jigged or teetering beds.

Jigged or teetering beds have been developed by Weiss and Swinton (18, 19, 20, 21) to process solutions derived from crushed ores containing as much as 15 percent by weight of less than 200 mesh solids. The principle of the technique is to apply a high frequency pulse to beds of solids to give them characteristics akin to fluidized solids but without the solids mixing of normal fluidization.

The original idea used a mineral jigging screen to apply the high frequency pulse but this required repeated pumping of the liquid stream. Two variants were devised. In the simplest form (21), a packed bed of solids was hydraulically pulsed and jigged solids could easily be removed from either the top or bottom of the bed. The lack of relative motion of the particles

was shown by comparing the output of the jigged bed with the output when it was operated as a packed and as a normally fluidized bed (19). No solids were withdrawn or added during these comparisons. The second variant (18) used a column with screens inserted at intervals and each of these supported a discrete bed of solids. Solids overflowed through downcomers.

The second variant seems to have been devised to increase the liquid throughput of jigged beds. This is the main disadvantage of jigged beds since the liquid velocities must be sufficiently low to avoid normal fluidization and mixing of solids. However, this disadvantage may be unimportant in certain difficult processes. For example, it was shown that the yield of streptomycin in its culture broth could be increased fifty-fold but that the advantage was offset by the difficulty of filtering the more fibrous broth prior to extraction of the streptomycin onto ion-exchange resin. A teetering bed was able to handle this broth and eliminate the filtration step (22).

1.2.4. Miscellaneous techniques.

Two other techniques capable of handling fines in the liquid must be mentioned. Morris (23, 24) designed

a mixer-settler in which solids and liquid flowed counter-currently through specially designed channels between a series of horizontal cells. The vigorous stirring in the mixer-settlers would be expected to cause serious attrition of ion-exchange resins and is said to give very low efficiency of solids-liquid contacting (17).

In the "resin-in-pulp" technique (25), resin was contained in a series of perforated baskets which were vibrated to improve contact with the liquid in which they were immersed. The contacting was cyclic and there was no discharge of resin from the apparatus. Continuity of operation and countercurrent flow for each of the several processes carried out during the cycle was attained by moving the liquid feed points. This is a promising technique where fines must be handled though it is rather complicated.

1.2.5. Fluidized beds.

Fluidized beds would seem to give the advantages of jigged beds and also allow higher liquid throughput. Multistage techniques are required to give countercurrent contacting and such were devised by Perry (26) and by Turner and Church (27). These use columns which are very similar to Weiss and Swinton's second variant. Since the downcomers through which the solids pass end below the

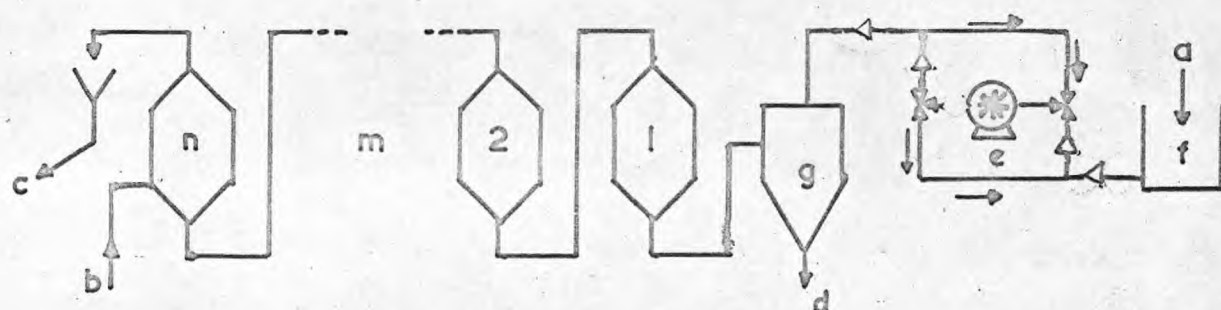
surface of the next lower fluidized bed, liquid bypassing through the downcomers is insignificant. However, this situation does not obtain at start-up when the column is empty of solids and the column appears to be difficult to start-up and rather unstable to operate.

It was hoped that the technique investigated in this study would be able to apply multistage fluidization with completely stable solids hold-up. In contrast to the developments outlined in section 1.2.4, the new technique appeared to be comparatively simple and free from moving parts in the processing section.

1.3. The technique investigated.

Fig. 1 shows a flow sheet of the technique in its simplest form. Essentially, this consists of a number of stages through which there is an overall countercurrent flow of solids and liquid. In practice, it was considered to be desirable to use a rather more complicated type of stage of the type shown in fig. 12. The instantaneous liquid flow is cyclic (fig. 2) and in the first part of the cycle, liquid flows in the forward direction fluidizing the beds of solids. There can then follow a period of no flow to allow solids to settle. Settling is not essential if the fluidized bed settles in less time than that of reverse flow. Next, reverse flow carries solids in dense phase flow from the contactor to the retainer from which a proportion is overflowed to the next contactor. Finally, forward flow is resumed and the solids in the retainer are returned to the contactor from which they originated. The average rate of liquid throughput is given by the difference between the forward flow volume, F , and the reverse flow volume, Q , divided by the total cycle time, t_c . Similarly average solids throughput rates can be obtained if the solids throughput per cycle is known. Since the volume of solids transferred is independent of the forward flow volume, the

FIG. 1 - FLOW DIAGRAM OF MULTISTAGE CONTACTOR.

Key.

- a - liquid feed
- b - solids feed
- c - liquid output
- d - solids output

e - liquid feed pump and flow reversing valves

f - liquid feed tank

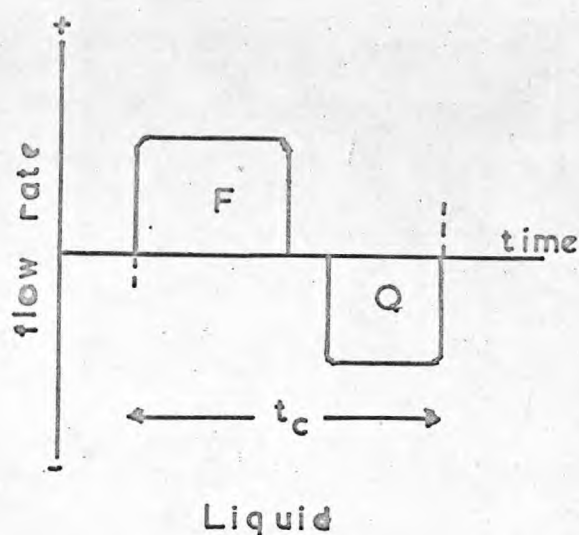
g - solids-liquid separator

m - contacting stages 1 - n.

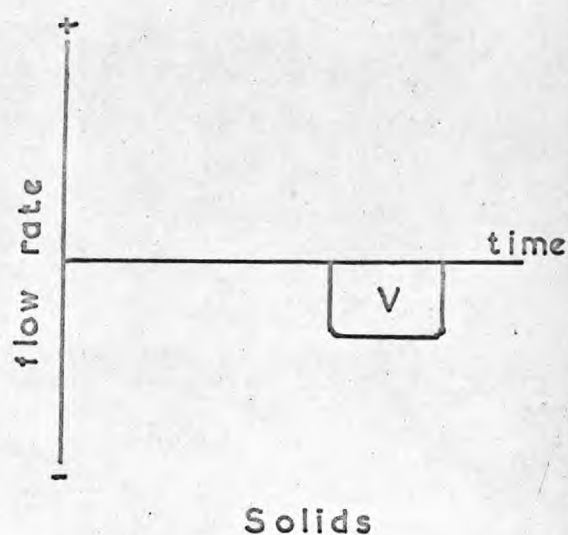
→ forward flow

← reverse flow

FIG. 2 - GRAPHICAL REPRESENTATION OF FLOW CYCLE



$$\text{Throughput rate} = \frac{F - Q}{t_c}$$



$$\text{Throughput rate} = \frac{V}{t_c}$$

Key.

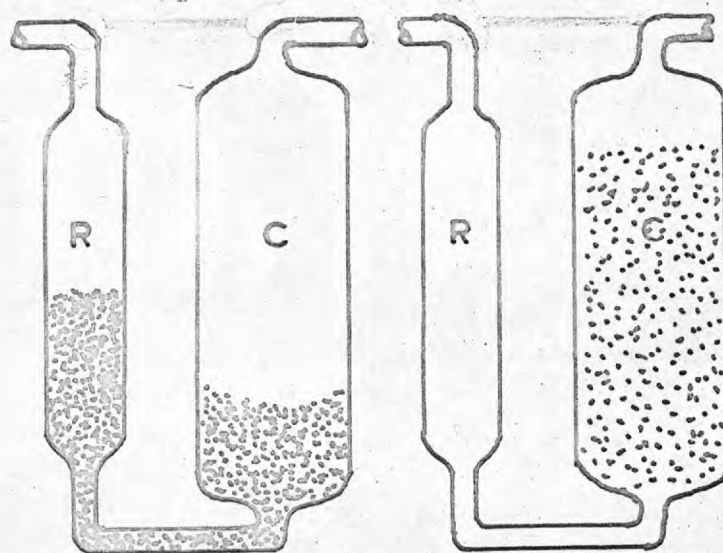
F - forward flow volume

Q - reverse flow volume

V - transferred solids volume

+ forward

- reverse

FIG. 12.CONTACTOR STAGES.

Reverse flow

Forward flow

Key.

R — Retainer
C — Contactor

ratio of solids to liquid throughputs is variable from zero to infinity in any apparatus by adjusting only the forward and reverse flow volumes.

The operation of the unit is controlled by these volumes of forward and reverse flow and they can be regulated by altering the direction of flow to the apparatus with the three-way valves before and after the pump. The valves can be operated by volume measurement using float switches in the liquid feed tank or, if a positive displacement pump is used, by a cycle timer.

The horizontal arrangement of the stages in fig. 1 is arbitrary and vertical arrangement in a column would function similarly.

1.4. The Scope of the Experimental Investigation.

Part I of this study is concerned with the hydrodynamics of a contactor using this technique. The dense phase flow of solids in liquid was sufficiently important and unusual to warrant separate investigation in Part II.

The hydrodynamic study of the contacting technique was focussed on the reverse flow part of the operating cycle since fluidization has been widely studied. Experiments on reverse flow were designed, firstly, to prove the operating feasibility of the technique and,

secondly, to investigate and correlate the parameters controlling solids movement in the apparatus. To achieve the first objective, it was necessary to show that solids hold-up would be stable throughout a multi-stage unit when the controlling parameters were suitably arranged.

A decision had to be made at this stage as to whether the effect of retainers should be investigated since they are not absolutely necessary to the technique. The main function of retainers was to give control and stability of solids hold-up in each stage during reverse flow. A variant of the technique - as in fig. 1 - could be envisaged which might have operated without retainers if an entire stage hold-up were transferred in a reverse flow period. However, retainers were essential for the transfer of smaller quantities of solids in a reverse pulse. Retainers were also expected to prevent any tendency for solids to move back through two stages in a single reverse flow period. Accordingly, the retainer was considered to be an integral part of the technique and investigation of reverse flow was confined to a design incorporating these.

The volume of a solids-liquid system is substantially independent of absolute pressure and the

behaviour of a single stage would be typical of the functioning of all stages in a cascade. Therefore, solids reverse flow was studied with only one typical stage. This greatly simplified accurate measurement of solids movement and allowed extension of the investigation to above the solids hold-up of normal multistage operation. Furthermore, a relatively large scale was possible with one stage without the necessity for large quantities of solids and the results could therefore be more relevant to industrial applications.

The systems investigated were silver sand - water and ion-exchange resin - water. Silver sand was used since it was cheap and was representative of a wide range of solids-liquid contacting processes such as the treatment of crushed ores. Ion-exchange resin was studied in later experiments since the technique offered advantages to several processes using this material.

2. EXPERIMENTAL.

2.1. Experimental equipment.

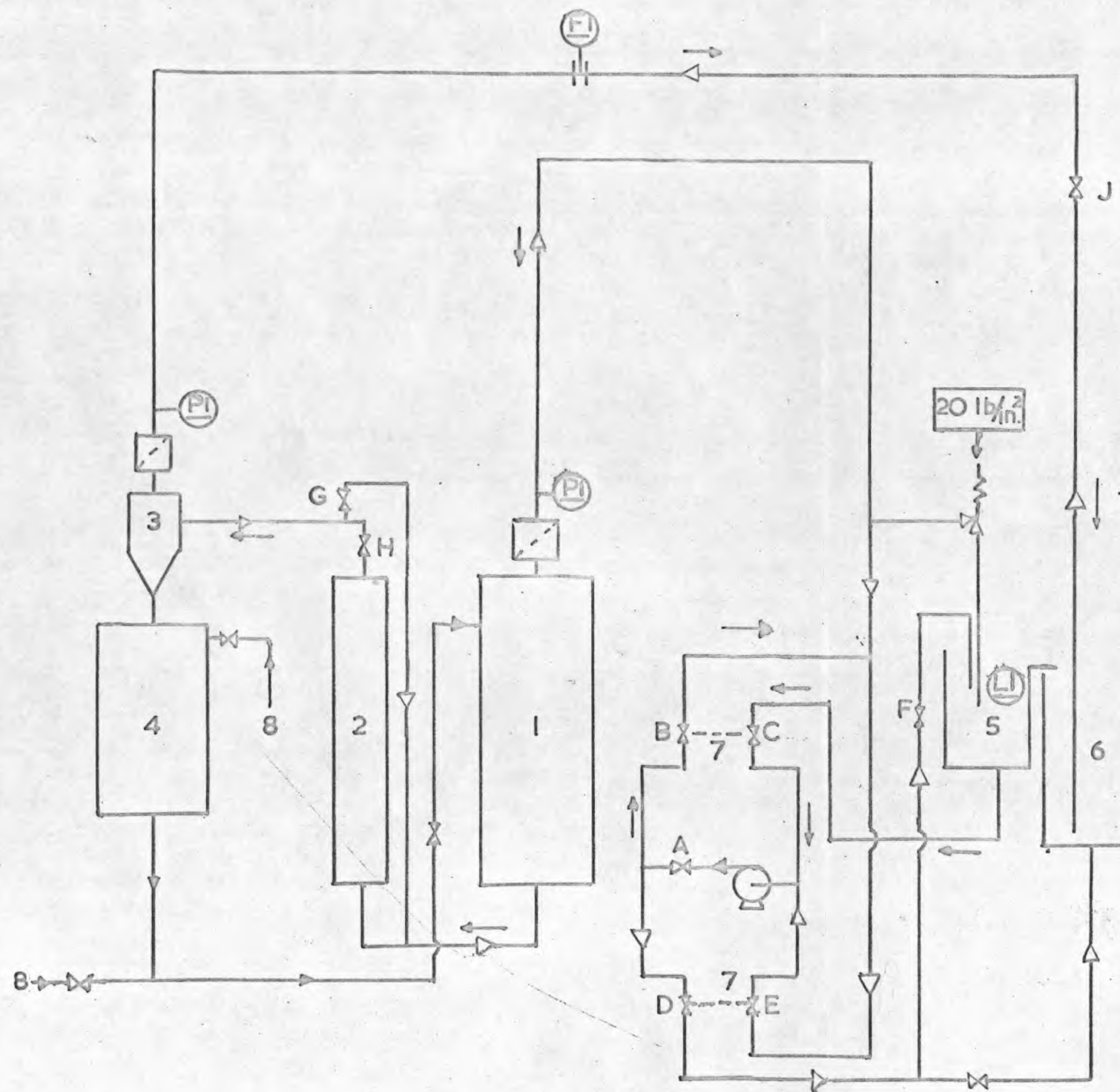
2.1.1. Experimental plant.

The flow sheet of the experimental plant is shown in fig. 3 and a photograph is shown in fig. 4.

The part of the apparatus representing a typical stage of a multistage contactor was the contactor and retainer and these are shown to scale in fig. 5. This unit was fabricated in standard Q.V.F. parts (Q.V.F. Ltd., Stoke-on-Trent, England). The contactor was constructed from two similar glass sections of 12 inch nominal diameter. A rigid polyethylene 60° cone (Baird and Tatlock Ltd., London, England) was fitted in the lower section with the cone outlet fitting tightly into the base outlet from the glass vessel. The lip of the cone fitted tightly between the two glass sections. Hence, this cone and the top glass section effectively formed the contactor. The contactor had a capacity of about 26 litres. The top outlet from the contactor was fitted with a candle filter to prevent the escape of solids.

The retainer was constructed from a 12 inch long by 6 inch diameter pipe section joined by two 8 inch long reducing sections to the 1 inch diameter

FIG. 3 - FLOW SHEET. OF SINGLE STAGE CONTACTOR.



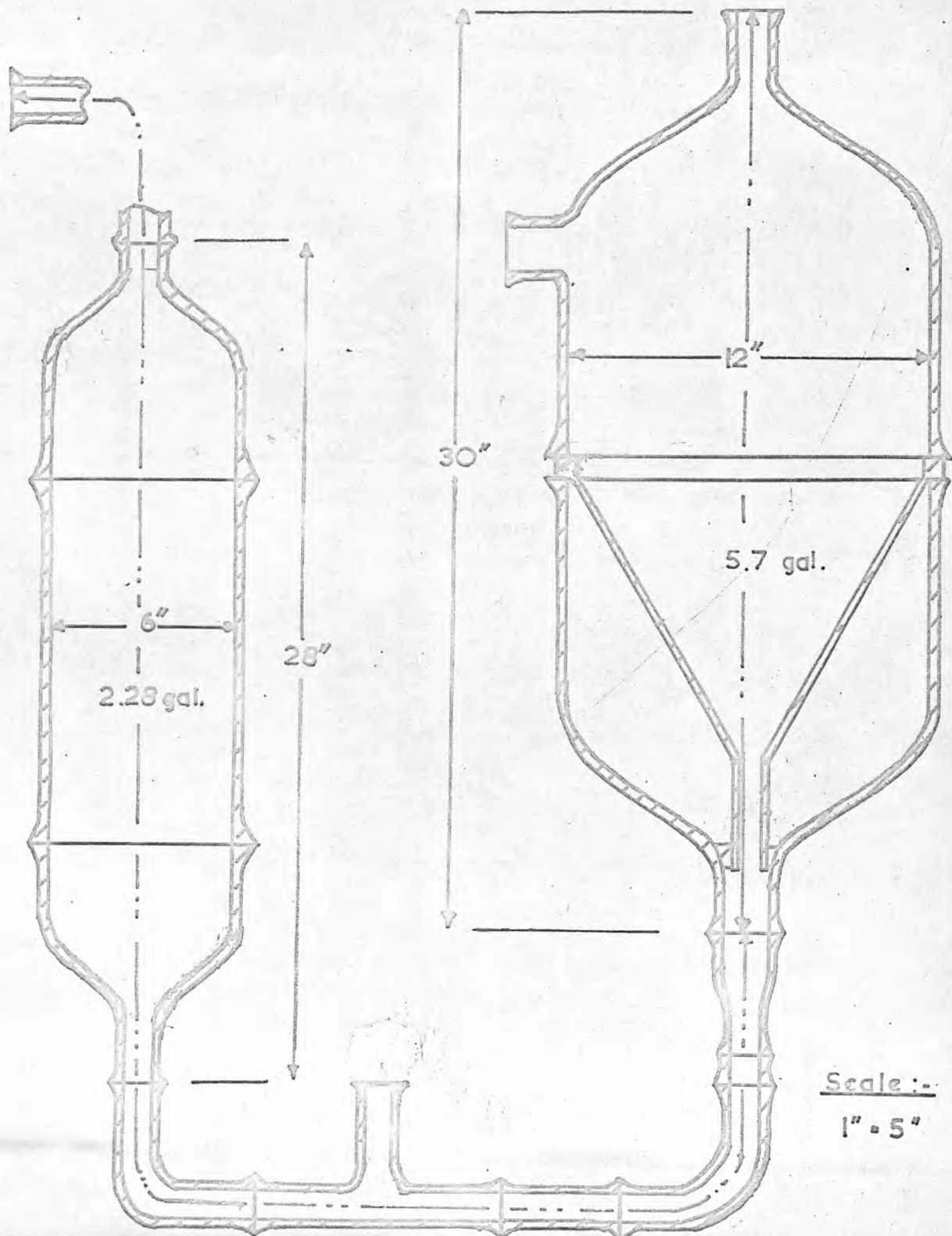
KEY.

- 1 - contactor
- 2 - retainer
- 3 - cyclone
- 4 - receiver
- 5 - metering tank
- 6 - main tank
- 7 - link between valves
- 8 - mains water input
- forward flow
- reverse flow
- Valve letters refer to text.



Fig. 4 Photograph of single stage contactor

AXIAL SECTION THROUGH CONTACTOR AND RETAINER



transfer lines. The capacity of the retainer plus transfer lines was 10.35 litres.

To allow solids to be completely and easily transferred out of the contactor, a 1 inch line was fitted to bypass the retainer. All the lines between the contactor, the retainer, and the cyclone were 1 inch nominal bore and 1 inch bore cocks were fitted to divert flow through the bypass (valve G open) or through the retainer (valve H open). Standard Q.V.F. glass was used throughout this section except for a short length of 1 inch flexible P.V.C. line just before the cyclone.

The cyclone and the receiver were standard spherical Q.V.F. vessels of approximately 13 and 20 litres capacity, respectively. Again, a candle filter was fitted to prevent solids from being carried beyond the cyclone. This line carrying water from the cyclone also passed through a short section of 1 inch flexible P.V.C. line.

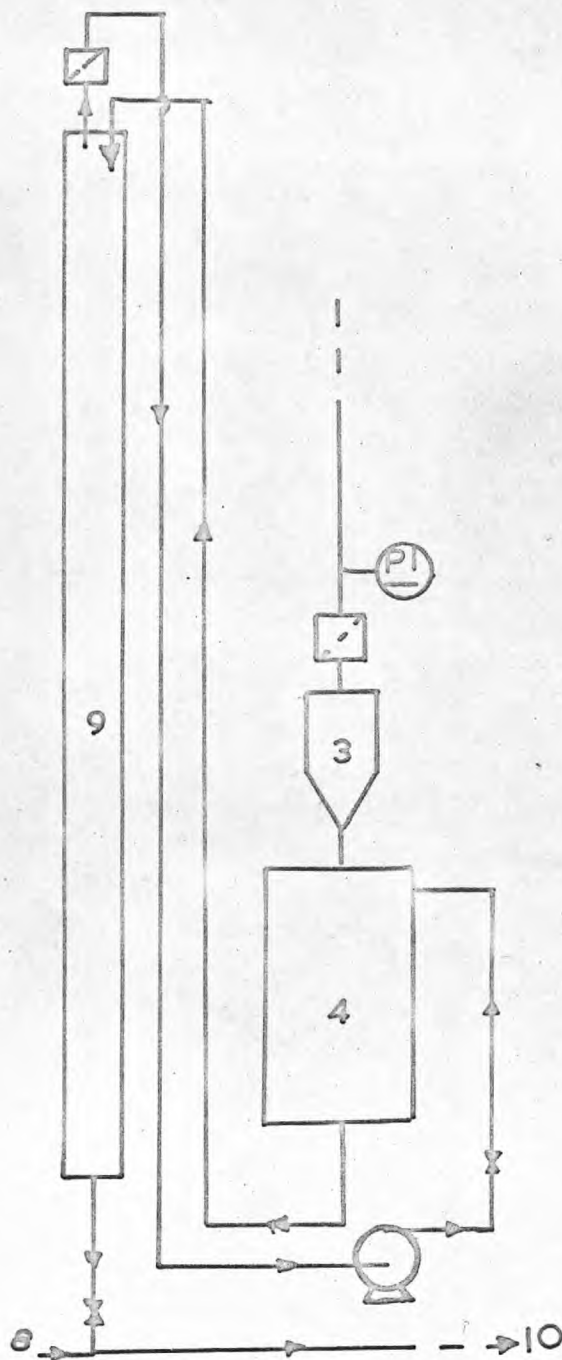
Solids could be transferred from the receiver to the contactor by appropriate isolation of the cyclone-receiver assembly (valves G, H and J closed) and pressurising it with mains water to eject solids through the $\frac{1}{2}$ inch polyethylene line from the base of the receiver

to the contactor. Dense phase flow of solids in this $\frac{1}{2}$ inch line proved to be difficult to achieve with the 20 lb./in.² pressure limit on the large glass vessels and a small water bleed was added close to the outlet from the receiver.

The volume of sand in the receiver was determined by weighing the cyclone-receiver assembly - hence the flexible links in the lines to the cyclone. Weighing was done with a spring balance capable of weighing 200 lb. This balance could be loaded or unloaded by moving it up and down using a reversing geared electric motor.

The small density difference between ion-exchange resin and water made weight determination of solids volume unacceptably insensitive. Instead, the solids in the receiver were transferred to a metering column. This was a 4 inch diameter by 8 foot long pipe section of about 20 litres capacity. A flow sheet of the modification is shown in fig. 6. Solids were pumped from the bottom of the receiver in dense phase flow through a $\frac{1}{2}$ inch P.V.C. line by a small centrifugal pump operating on a closed loop between the receiver and the top of the metering column. With the velocities used, the solids disengaged by gravity (at the top of the metering column) from the circulating

FIG. 6 - FLOW SHEET OF MODIFICATION
TO FIG. 3 TO MEASURE RESIN.



Key.

- 3 - cyclone
- 4 - receiver
- 8 - mains water
- 9 - metering cylinder
- 10 - resin to
contactor

flow. As a precaution, a screen filter was fitted across the entry to the return $\frac{1}{2}$ inch P.V.C. line. The circulating pump was a small centrifugal.

Flow in the main loop through the contactor, retainer, and cyclone was produced by a centrifugal pump with an output ranging from 850 gal./hr. against a 45 foot head of water to 2000 gal./hr. against a 5 foot head. The direction of flow around the loop could be reversed by appropriate operation of valves B, C, D and E. The velocity of reverse flow was controlled by valve A. Forward flow velocity was controlled by valve F. (Although forward flow passed through both valves A and F, reverse flow velocity was always at least double that of forward flow.) A safety valve was fitted between the pump and the contactor and set to open at 20 lb./in.² to protect the 12 inch glass vessels.

The lines between the main pump, the tanks, the cyclone, and the contactor were all of 1 inch nominal bore galvanised steel except for the section containing valve F which was $\frac{1}{2}$ inch nominal bore.

The tanks were made of galvanised steel sheet. The capacity of the main tank was approximately 50 litres and that of the metering tank about 25 litres.

Forward flow could be continued indefinitely with water overflowing from the metering tank to the main tank. With reverse flow, water was withdrawn from the metering tank and delivered to the main tank.

2.1.2. Characterisation of the solids materials.

2.1.2.1. Silver sand.

The silver sand was initially screened by a Russell screen to a nominal -36+52 B.S.S. mesh size. Fig. 7 shows a sample of the screened sand with a magnification of $\times 14$. On a similar photograph the sizes of 175 particles were measured to the nearest $\frac{1}{4}$ cm. (equivalent to an actual size interval of 0.018 cm.) using a graduated perspex "V". The statistical diameters technique of Martin (32) was followed and the "diameter" of a particle was measured as the projected length on a fixed axis of a line estimated to bisect the visible area of the particle. The number found of any given statistical diameter was then multiplied by the cube of the statistical diameter to give an estimate of the weight of that fraction and the cumulative estimated weight was tabulated against particle size in the conventional manner of size distribution characterisation.

The absolute density was found to be 2.646 gm./ml. from density bottle determination. The free-settled voidage was measured by weighing a measured volume of sand and water and was found to be 0.489 while the tap settled voidage was 0.383. The fluidization characteristics were observed directly by measuring the pressure drop over a bed

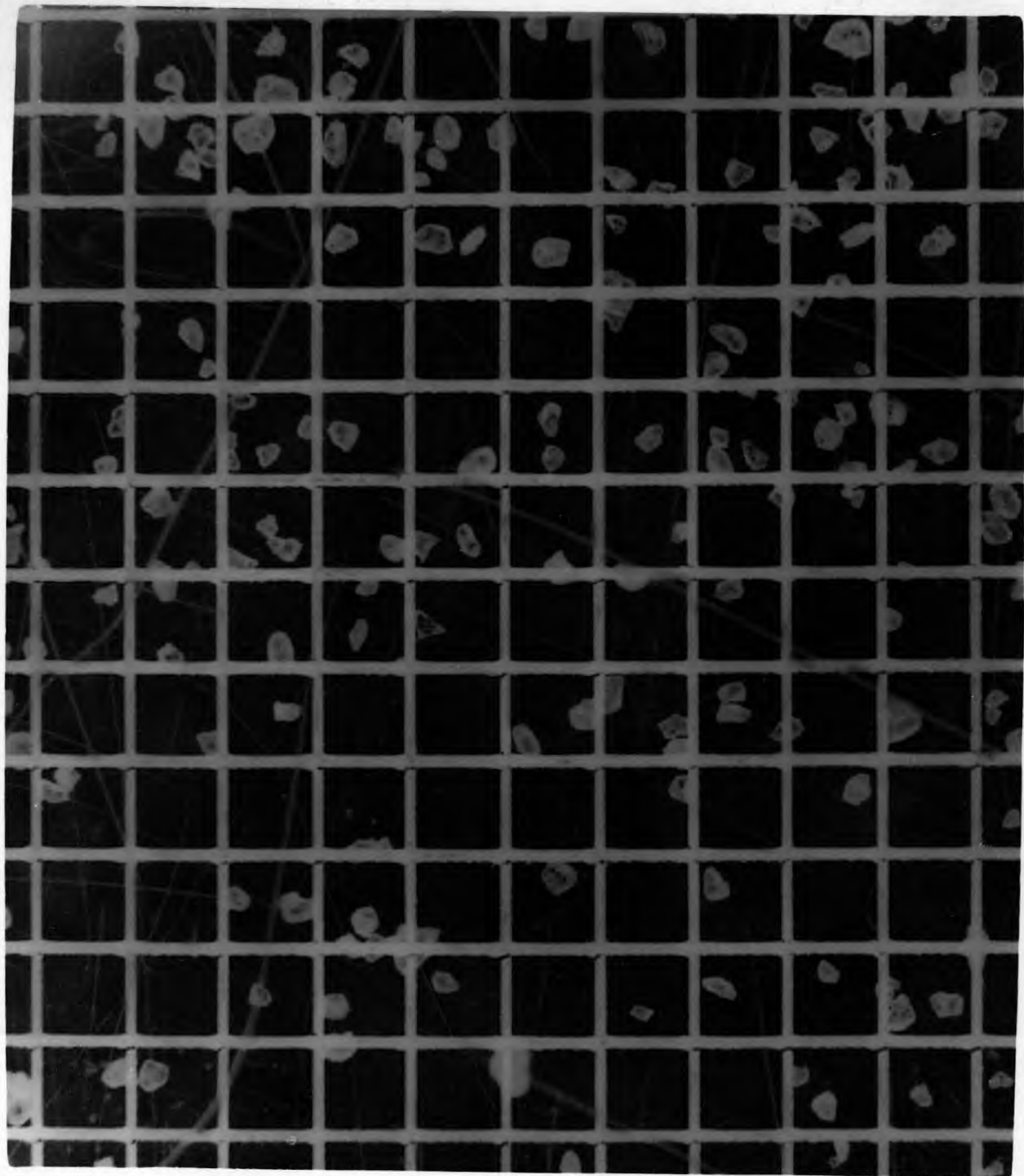


Fig. 7. Silver sand, magnification x 14. 1 mm. grid.

of solids in a 2 inch diameter column in which the liquid flow was distributed by a cloth distributor. The details of fluidization and size distribution are given in Appendix 1.

2.1.2.2. Ion-exchange Resin.

The ion-exchange resin was Zeocarb-225, 8% D.V.B. cross-linked, sodium form, -14+52 B.S.S. mesh. (The Permutit Co. Ltd., London, England.) Figures 8 and 9 show the resin magnified by x 13. Figure 8 is resin in unused condition and figure 9 shows resin which had been used throughout the experiments. One hundred particle diameters were measured to estimate the size distribution in the same way as for silver sand. Spheres could be measured without recourse to statistical diameter techniques and fewer measurements were considered necessary. The absolute density of resin which had been used throughout the experimental work and from which adhering water had been removed by centrifuging was 1.295 gm./ml. which was not significantly different from the density of the unused resin which had been equilibrated in distilled water for 24 hours. This had a density of 1.297 gm./ml. This is not unexpected since the principal ion absorbed from the mains water used in the experiments would be calcium with

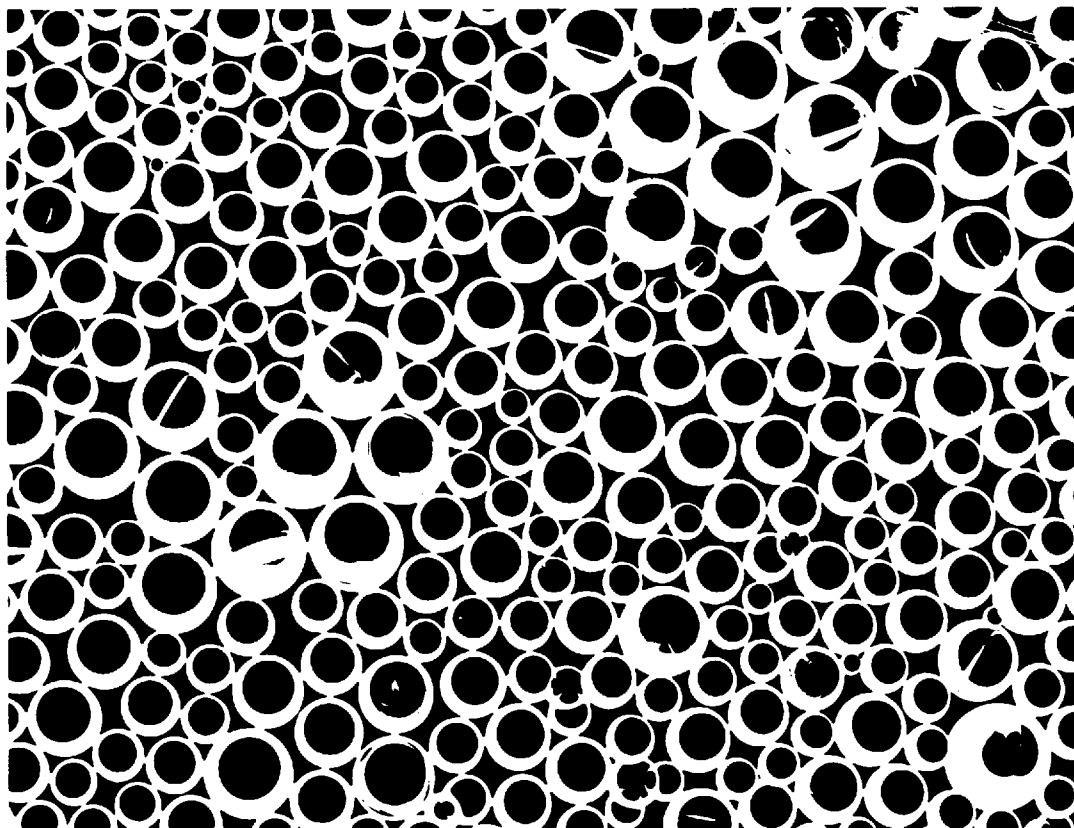


Fig. 8. Unused ion-exchange resin.

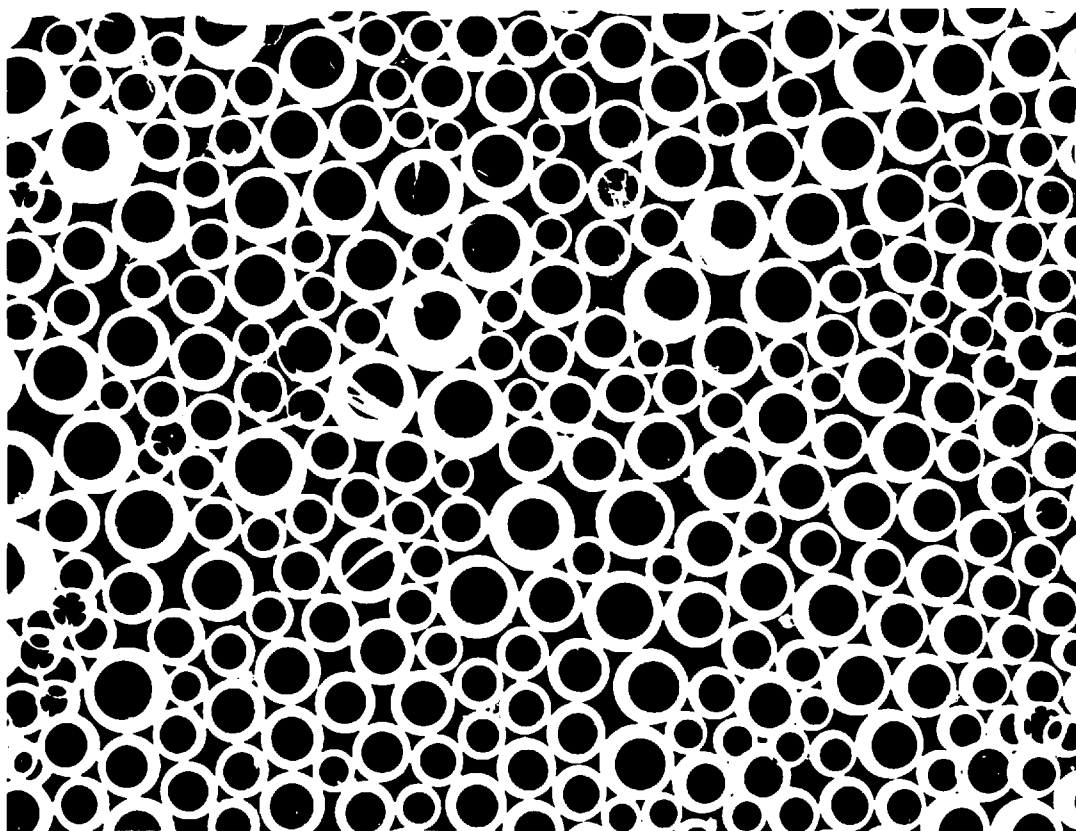


Fig. 9. Ion-exchange resin used throughout the experiments.
Negative photographs, magnification $\times 13$.

an equivalent weight of 20 compared to that of sodium of 23. The free-settled voidage was 0.325. Full details of characterisation of fluidized resin and size distribution are given in Appendix 1.

2.2. Planning of experiments.

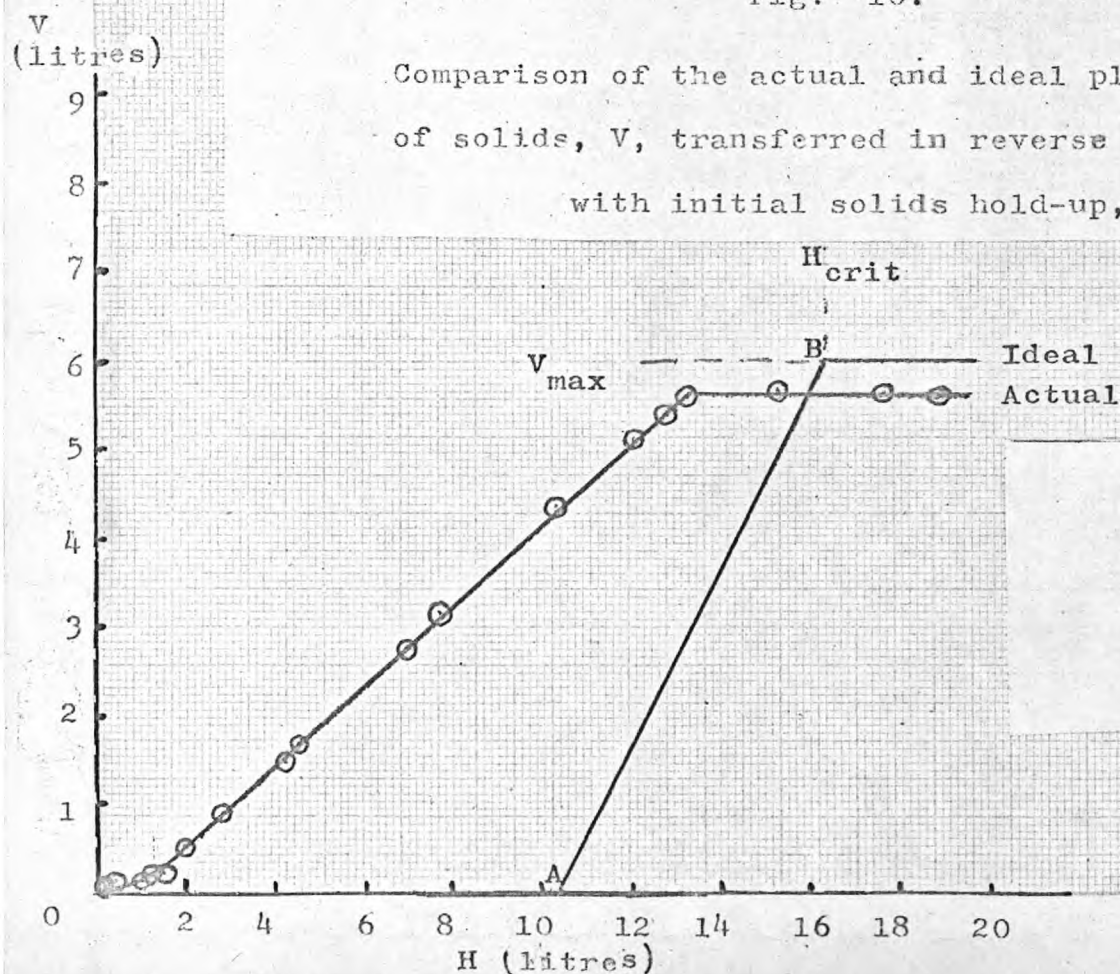
2.2.1. Dependent variables.

Figure 10 shows diagrammatically an idealised plot of the volume of solids, V , transferred out of a stage in one reverse flow volume as a function of the independent variable of initial stage hold-up, H . This plot would arise at very low flow rates which were insufficient to fluidize the solids in the retainer. The volume of solids represented by OA would fill the retainer and if the hold-up of solids was less than or equal to OA , no solids would be transferred beyond the retainer regardless of the size of the reverse flow volume, Q . With hold-ups of solids greater than OA , the excess of the hold-up over OA would be transferred out of the stage until the hold-up equalled the reverse flow volume. This would give rise to the AB section of the plot with a gradient of unity. Further increases in hold-up beyond B would remain in the contactor throughout reverse flow and the volume of solids transferred would remain constant.

Before the full experimental programme was planned, some preliminary experiments were made to determine the general characteristics of the results. An actual plot of V versus H with the much higher reverse flow rates used in the experiments is also shown in fig.10. This illustrates the choice of the dependent variables.

Fig. 10.

Comparison of the actual and ideal plots of volume of solids, V , transferred in reverse flow volume, Q , with initial solids hold-up, H .



Material - ion ex. resin

Flow rate - 30.3 l./min.

Settling time - 0 sec.

$Q = 16.3$ litres

The selected dependent variables were:

- (1) $V_{\max}$, the greatest volume of solids which could be transferred out of the stage;
- (2) H_{crit} , the lowest initial solids hold-up to give a solids transfer out of the stage of $V_{\max}$;
- (3) U , the retainer utilization. That is, the free-settled volume of solids in the retainer at the end of reverse flow with an initial hold-up of H_{crit} , expressed as a fraction of the retainer volume
- (4) m , the operating gradient. That is, the slope of the section of the graph where V increases with increasing H .

Considering the choice of these variables in detail, $V_{\max}$ was of obvious importance since it must be known to determine the solids throughput of a contactor. Departure of $V_{\max}$ from the idealised case might be expected to arise partly due to an absence of settling time before reverse flow and partly due to the effect of slip. In moving solids by the drag force of a flowing liquid, the liquid must move faster than the solids and the excess liquid passing is referred to as slip.

To show the significance of H_{crit} , consider two stages of a multistage unit of which one is the solids feed stage. The feed stage has an adequate supply of solids and provides the only source of solids to the other stage. Assuming that initially the dependent stage contains only liquid and that the two stages go through a number of idealised flow cycles, the dependent stage will acquire more solids from the feed stage than it discharges until the hold-up in the dependent stage is H_{crit} . H_{crit} is the normal operating hold-up for a stage.

While the dependent stage is filling with solids, reverse flow out of this stage ends with a period of dilute phase flow. V_{max} is reached when all this dilute phase flow is replaced by dense phase flow. However, at V_{max} dilute phase flow will be on the point of replacing dense phase flow out of the stage and dilute phase flow will have entered the retainer some time before. The retainer utilization, U , is a measure of the volume of diluting water in the dilute phase entering the retainer. U is useful in determining the actual volume of a retainer to hold back a desired volume of solids through a reverse flow period.

The operating gradient, m , is an indicator of the variation of solids transferred, V , with solids hold-up, H ,

when the latter is less than H_{crit} . With ion-exchange resin, the variation was linear over most of the hold-up below H_{crit} . In some cases with the silver sand, the variation was less uniform. For the purposes of statistical analysis in such cases, a gradient closely representative of at least two-thirds of the sloping section was used.

2.2.2. The factorial experiments.

2.2.2.1. Factorial experimentation.

To assess the effects on the statistics of the independent variables, a factorial experiment was devised. This form of experimentation was pioneered by Fisher and his co-workers (28, 29) in the Nineteen-thirties during their work in agricultural research.

For many years, experimental work in chemical engineering was carried out by the classical method of studying a dependent variable by varying one independent variable and holding all the other variables constant. This has many disadvantages. No idea of the accuracy of the experiment could be derived from the results unless experiments were replicated. Uncontrolled variables could profoundly affect the results - for example, in the present experiments, solids breakage could perhaps have caused a significant effect. And, finally, no information on the effects of interactions between the variables could be derived.

In a factorial experiment, the effects of uncontrolled variables are allowed for and largely eliminated by randomisation of the experiments. No experiments need be replicated since information on the effect of experimental

error can be derived from apparent interactions of small magnitude and statistical non-significance. Of course, this method would fail if almost all the interactions were significant but this is unlikely and in any case the classical method conceals the very existence of interactions. Finally, factorial experimentation is superior in interpreting all but the most definite effects of independent variables in that factorial analysis provides an estimate of the probability that a measured effect is real and does not arise by random chance.

Factorial analysis is not a technique which is universally applicable since it cannot deal adequately with results where there is a sharp discontinuity in the effect of one of the independent variables (or factors, in statistical terminology) on the dependent variable (or statistic). Effective factorial analysis is also limited by the requirements of stringent randomisation of experiments and equal spacing of factorial levels. The errors in the statistics derived from the factorial experiment must be normally distributed which would not occur, say, if the error in making measurements varied markedly across the range of levels of one factor. However, non-normal statistics can frequently be transformed

into a normal distribution and even with analysis of non-normal data, the conclusions can usually be accepted unless the departure from normality is very severe or the significance of a conclusion very marginal.

It must be emphasised that the results of factorial analysis should be examined critically since there is always a small probability that an apparently significant result may have arisen by random chance. Especial scepticism is advisable if the residual error is much smaller than the anticipated error. Factorial analysis is particularly useful in dealing with statistics which are influenced by a large number of variables.

2.2.2.2. Design of the factorial experiments used.

To allow full randomisation, a factorial experiment must be planned fully before any experimental work is performed. Investigations of the effects of factors (independent variables) on the reverse flow of solids in the present work could have included reverse flow rate, forward flow rate, settling time, reverse flow volume, retainer volume and shape, and initial stage hold-up. An experiment covering all these variables would have been excessively large.

Solids hold-up could not be included in the experiment since its effect was sharply discontinuous

at H_{crit} . Therefore, it was decided to make each experiment of the factorial design include a series of runs over the full range of initial solids hold-up to obtain the dependent variables discussed in section 2.2.1. and to use these variables as the statistics of four simultaneous factorial experiments.

Retainer volume and shape were not included in the experiments. Nothing was known of how changes in the retainer design might affect the statistics and it was not considered important enough to justify the complications of alternative retainers in the experimental equipment.

Forward flow rate was not included since its effect on settling time would be expected to be predictable. An increased forward flow rate would be expected to increase the magnitude of any difference between runs with no settling time and those runs with full settling of solids before reverse flow.

The remaining factors; reverse flow volume, Q , reverse flow rate, R , and settling time, T , were selected for investigation. Settling time was set at two levels - no settling, or substantially complete settling which was taken as 50 seconds. Reverse flow rates of 30.3 and 60.6 litre/min. were used for silver sand and of 30.3 and 45.4

litre/min. for ion-exchange resin. A high pressure drop with ion-exchange resin made 60.6 litre/min. unobtainable. Three levels of reverse flow volume were used of 13.1, 16.3, and 19.5 litres. This gave a $2 \times 2 \times 3$ factorial design of twelve experiments - one each for every combination of the variables.

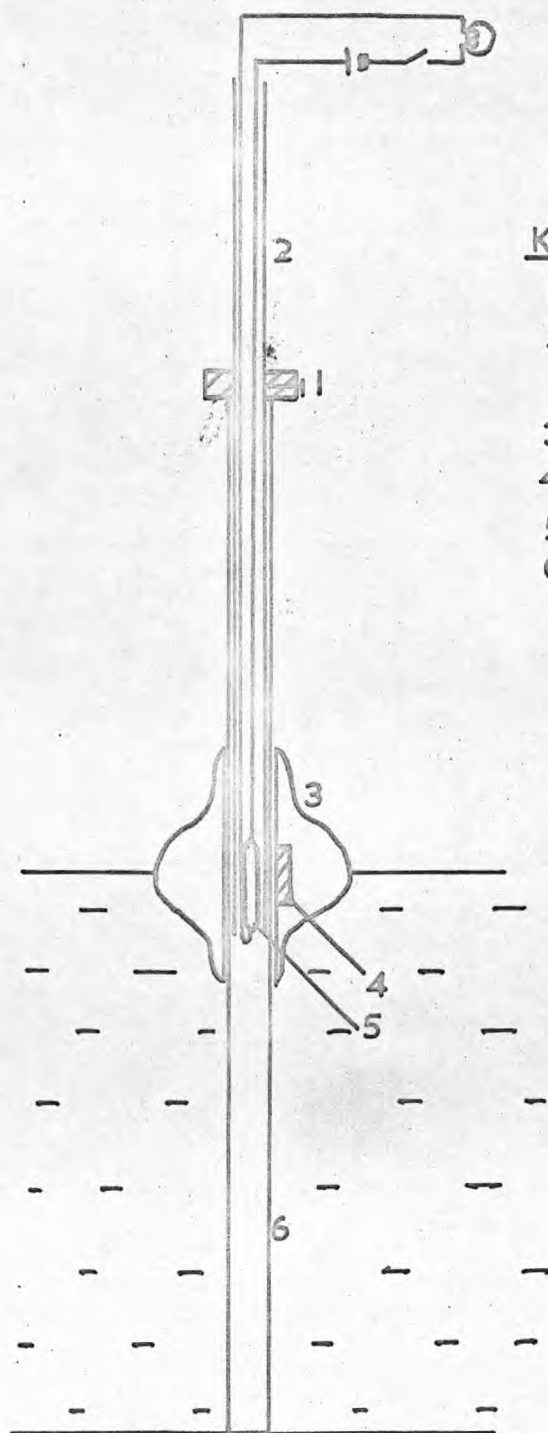
Reverse flow rates were set before a series of experimental runs using the main throttling valve, A (fig. 3). Forward flow was set at a fixed rate throughout of 15.3 litre/min. This was lower than would normally be used in practice but enabled the contactor to be loaded with solids to far beyond the normal operating volume without large quantities of solids collecting on the candle filter at the top of the contactor.

It would have been desirable to use one $2 \times 2 \times 3 \times 2$ design of experiment including the solids material as the fourth factor. However, to inter-bandomise the two sets of experiments would have required considerable enlargement of the apparatus and elaborate precautions would have been required to prevent intermixing of the two solids which would have been most undesirable on even the smallest scale. So two factorial experiments were carried out in succession for silver sand and ion-exchange

resin.

The details of factorial analysis are given in Appendix 2.

FIG. II—FLOAT SWITCH IN METERING
TANK.



Key.

- 1 - locking collar
- 2 - movable inner tube
- 3 - float
- 4 - magnet
- 5 - reed switch
- 6 - fixed outer tube

2.3. Experimental methods.

2.3.1. Measurement and control of variables.

2.3.1.1. Volume of reverse flow.

The volume of reverse flow had to be accurately controlled as well as accurately measured. The change in level in the metering tank could be followed on a carbon tetrachloride-water manometer but there was a marked lag in operation and it could not be used to give reproducible reverse flow volumes. After some moderately successful attempts to produce an instantaneous indication of a particular level by using a mercury float switch, a reed switch device was fitted and gave excellent reproducibility. The reed switch is a magnetic proximity switch which can make an electrical circuit. The maximum distance at which a particular magnet closed the contact reeds was found to be substantially constant.

In its use as a float switch, the reed switch was mounted in a brass tube which was graduated in 1/20 inch units. (Fig. 11.) Using these graduations, this tube could be positioned inside another wider brass tube fixed in the metering tank. A small permanent magnet was mounted inside a hollow polyethylene float which slid up and down the fixed brass tube. When the proximity of the magnet closed the

reed switch, the circuit through a small light was completed and the reverse flow of the pump was stopped by manually operating one pair of linked valves (B and C) controlling the direction of flow round the main pump. This gave excellent reproducibility of reverse flow volume. Reverse flow was always started with the metering tank full. A small correction was applied to allow for the difference of levels when reverse flow immediately followed forward flow and when a settling period of no flow intervened.

The volume of reverse flow was set before each series of runs making up one combination of levels in the factorial experiment by location of the reed switch. For the experiments with silver sand, the required level in the metering tank was set by reference to the carbon tetrachloride-water manometer connected between the metering tank and a constant level reservoir. On analysis of the results for silver sand, it appeared that the volume setting must have been rather erratic and, for the experiments on ion-exchange resin, level settings were made by direct reference to the graduations on the rod containing the reed switch. This was completely satisfactory. Use of the carbon tetrachloride-water manometer should have given a level measurement of about double the sensitivity of direct measurement and the poor

quality of the results arose through the unnoticed effects of direct sunlight on the carbon tetrachloride which has a coefficient of expansion of 1.236×10^{-3} and could be observed visibly to expand if shielding from sunlight was removed. All manometers were subsequently shielded from direct sunlight. There was also a progressive tendency for carbon tetrachloride to cling to the manometer walls which was absent during initial testing.

2.3.1.2. Flow rates.

Flow rate in either direction through the system could be measured by a calibrated orifice meter located in the position shown in fig. 3. The orifice was made from 1/20 inch brass plate with a 0.72 inch diameter orifice and was corner tapped. The size of the orifice and its general design were based on British Standard specifications (30) though these were stated to be rigidly applicable for pipe diameters of 2 inches and upwards. However, the orifice was calibrated in situ and pressure drop was found to vary quadratically with velocity. This calibration was done by timing a fixed change in level in a 20 litre tank and the relationships were found to be $V = 461.7\sqrt{\Delta P}$ for forward flow and $V = 502.6\sqrt{\Delta P}$ for reverse flow,

where ΔP = pressure difference in cm. ethylene dibromide-water,

V = flow rate in litres/hr.

2.3.1.3. Settling time.

Small variations in settling time produced no significant effect on results and it was quite satisfactory to time the 50 seconds allowed for full settling with a wrist watch.

2.3.1.4. Solids hold-up and volume transferred.

The volume of solids could only be determined in the receiver (for silver sand) or in the metering column (for resin). Hence, hold-up was determined by measuring the total volume of solids in the appropriate metering vessel and the volume subsequently transferred to the contactor was found by difference from a second volume measurement. The volume transferred out of the retainer in reverse flow was determined by difference between this second measurement and a third measurement after the reverse flow period.

The absolute volumes of silver sand could readily be obtained by weighing since its density had been determined (section 2.1.2.). This was expressed as free-settled volume using the measured free-settled voidage. The absolute volume of ion-exchange resin was determined from observation of the change of levels in the metering column. Simple observation of the levels proved unreliable since the resin

tended to compact slowly on settling and rapidly if it experienced any vibration. It was found that reproducibility could be obtained by fluidizing the resin in the metering column with an approximately constant volume of water and measuring the level immediately after all movement of solids ceased. This was a direct measurement of the free-settled volume.

2.3.2. Operating details.

Before the experiments on reverse flow were begun, visual observations were made of the behaviour of a bed of solids particles during forward flow. This was observed to follow the pattern observed for single inlet gas fluidized solids beds with conical bases observed by Leva (31). With increasing liquid flow rates and increasing bed volumes, the behaviour of the bed moved from that of a spouted bed to a fairly evenly fluidized bed.

With the retainer full, it was found that solids would not flow in dense phase at rates of less than about 25 litre/min. at pressures tolerable in 12 inch diameter glass vessels. The upper limit, governed by the available pressure on the suction side of the pump, was found to be about 65 litre/min. for silver sand but when the experiments with resin were carried out it was found that the

upper limit was 50 litre/min. As the flow rate approached the lower limit, pressure rose rapidly, the safety valve opened, and flow ceased. The bypass line was used to clear such blockages since it was not difficult to apply sufficient pressure to move the stationary solids over the short section between the bottom of the contactor and the start of the bypass line.

With the limited pressure available on the suction side of the pump, a small water bleed through the bypass line was essential when returning solids from the retainer to the contactor at the end of reverse flow. Pressures of about 8 lb./in.² across the gland of the pump admitted air to the impeller chamber. By returning solids to the contactor from the retainer, a series of runs could be carried out with diminishing solids hold-up without having to transfer solids from the receiver to the contactor.

2.4. Experimental Errors.

The error in measuring the reverse flow volume, Q , was only about ± 0.04 litres. The quantity of solids transferred in a reverse flow volume could be measured to an accuracy of about ± 0.025 litres by weighing (silver sand) and to about ± 0.04 litres by volume measurement (ion-exchange resin). The stage hold-up, H , was measured to the same accuracies as the volume of solids transferred. To safeguard against any instrument drift, the silver sand inventory of the system as indicated by the spring balance was noted before and after each of the factorial runs. If the readings differed by more than ± 0.1 lb. (± 0.025 l.) the run was rejected and repeated. There was no purpose in measuring the settling time to a high degree of accuracy and this was only accurate to about one second. Similarly, the liquid flow rates did not require high accuracy adjustment and the manometer setting across the orifice plate would have allowed errors of ± 0.4 l./min.

3. RESULTS.

3.1. Nomenclature and terminology.

The experiments within the main factorial experiments are designated by the levels of the factors. Thus, $Q_3R_1T_2$ designates the experiment carried out with the highest reverse flow volume, Q , the lower reverse flow rate, R , and the longer settling time, T .

Effects of the factors are designated as X_m' . Thus Q_1' designates a first order effect on the statistic of the reverse flow volume, Q . If only two levels of the factor are investigated, X_1' would designate an effect of unspecified order. X_2' would generally denote a second order effect on the statistic of the factor. However, as only three levels of the reverse flow volume were used in these experiments, Q_2' denotes an effect of any order above first order.

Effects of interactions of factors are denoted as $X_m'Y_n'$, $X_m'Y_n'Z_o'$, etc. Thus, $Q_1'R_1'$ designates an interaction of reverse flow volume, Q , and reverse flow rate, R . A positive value of the effect of this interaction would mean that the effect on the statistic of increasing Q is numerically more positive at the higher level of R .

The levels of significance of effects are indicated in brackets. Thus, (1%) implies that the effect was significant with a probability of less than one percent that this could have arisen by random chance. The three main levels of significance are taken as 0.1%, 1%, and 5%. These levels are referred to as very highly significant, highly significant, and significant, respectively.

3.2. Results of the factorial experiments.

3.2.1. Factorial experiment on silver sand.

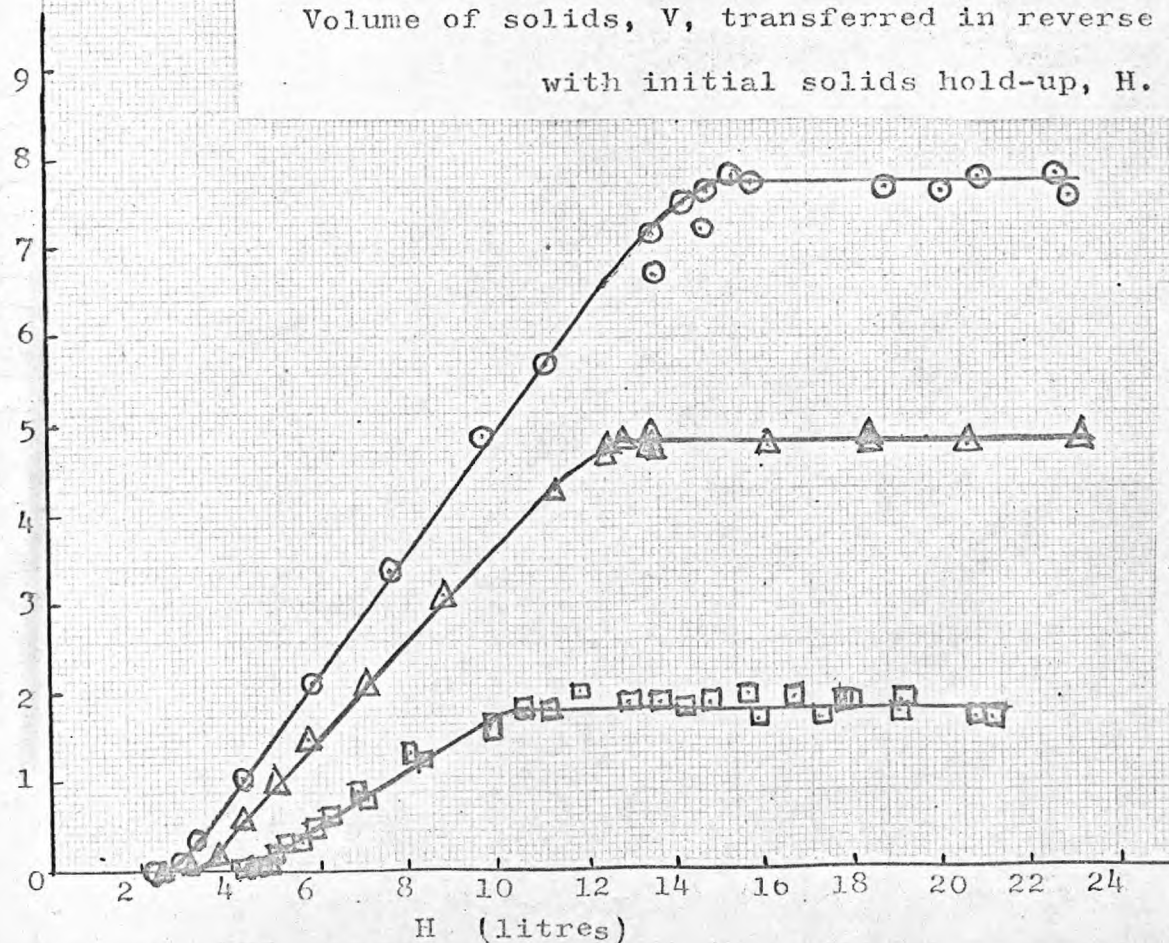
The results for the twelve experiments within the statistical experiment are shown in fig. 13 to 16.

There was a discernible difference in the shape of the plots for the two reverse flow rates, R . At the lower flow rate of 30.3 litre/min., there was a region of low initial hold-up, H , when no solids were transferred out of the stage during the reverse flow period. This gave a minimum stage hold-up which would be maintained indefinitely in the absence of solids feed. The minimum hold-up would not be maintained during continuous reverse flow as distinct from the normal cycling of forward and reverse flow. This is obvious, since this minimum solids hold-up decreased as the reverse flow volume, Q , was increased (fig. 13 and 14). At the higher reverse flow

V
(litres)

Fig. 13.

Volume of solids, V, transferred in reverse flow volume, Q,
with initial solids hold-up, H.



Material - silver sand

Flow rate - 30.3 l./min.

Settling time - 0 sec.

□ - Q₁ = 13.1 litres

△ - Q₂ = 16.3 litres

○ - Q₃ = 19.5 litres

Fig. 14.

Volume of solids, V , transferred in reverse flow volume, Q ,
with initial solids hold-up, H .

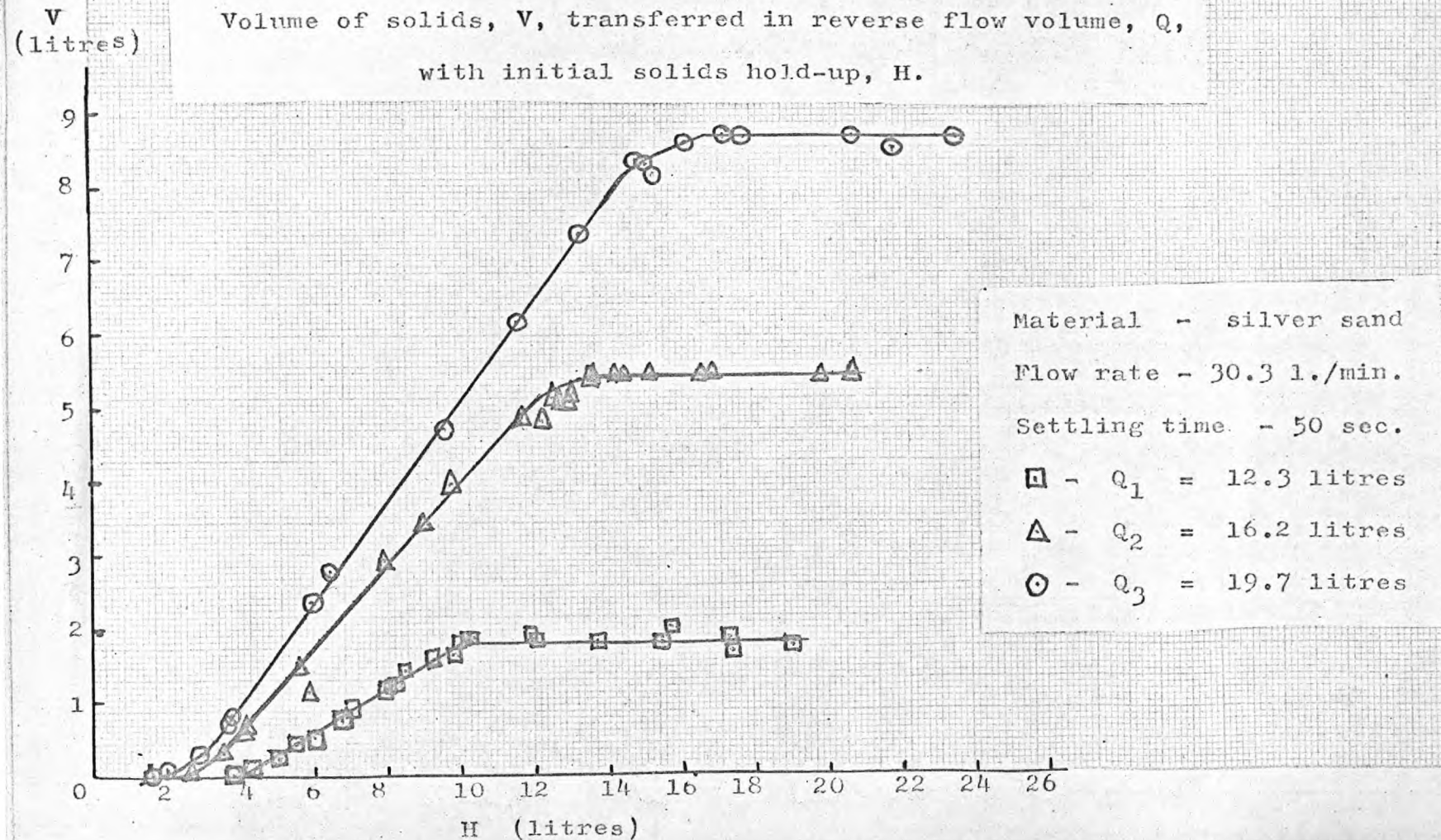
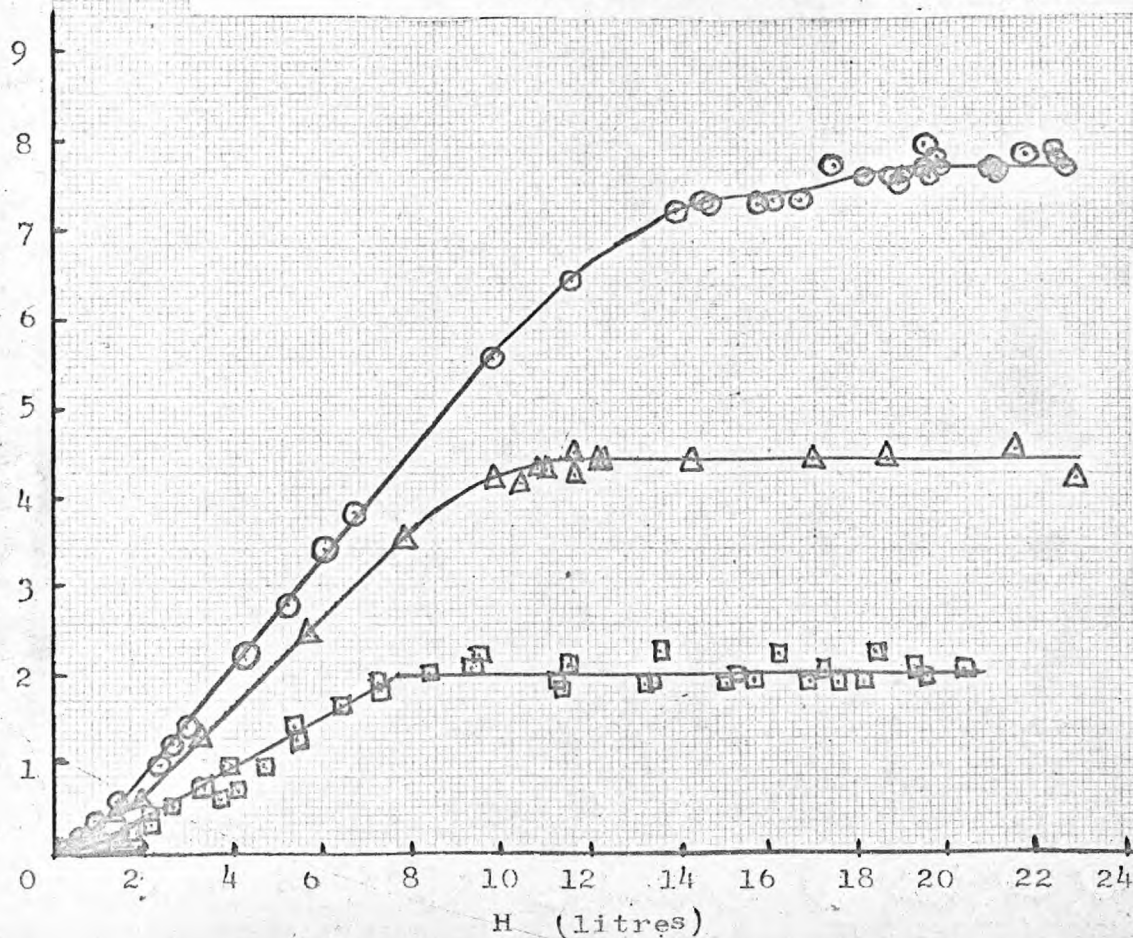


Fig. 15.

Volume of solids, V , transferred in reverse flow volume, Q ,
with initial solids hold-up, H .

V
(litres)



Material - silver sand

Flow rate - 60.6 l./min.

Settling time - 0 sec.

□ - Q_1 = 11.8 litres

△ - Q_2 = 15.3 litres

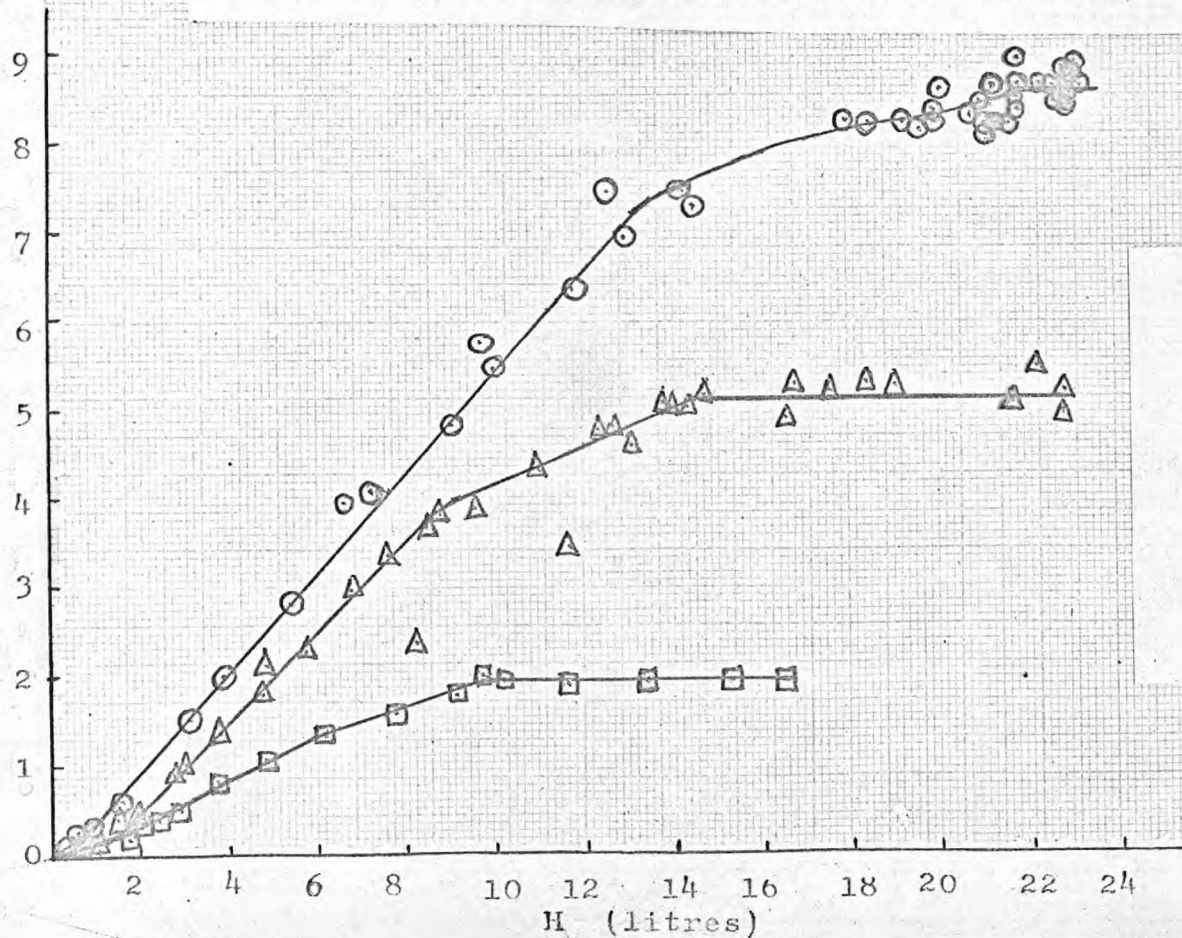
○ - Q_3 = 19.5 litres

H (litres)

Fig. 16.

Volume of solids, V , transferred in reverse flow volume, Q ,
with initial solids hold-up, H .

V
(litres)



Material - silver sand

Flow rate - 60.6 l./min.

Settling time - 50 sec.

□ - $Q_1 = 12.3$ litres

△ - $Q_2 = 15.7$ litres

○ - $Q_3 = 19.6$ litres

rate of 60.6 litre/min., the initial minimum hold-up volume did not occur though there was a slight curve in the plot close to the origin indicative of a tendency for the solids to be held selectively in the retainer.

The general structure of the plots was outlined in section 2.2.1. and analyses were made, as indicated in that section, of the maximum volume of solids transferred out of the stage, V_{max} , of the minimum stage hold-up, H_{crit} , giving a transfer volume of V_{max} , of the operating gradient, m , and the retainer utilization, U . The values of these statistics are listed in Appendix 2 with the factorial analyses.

The statistical analysis of H_{crit} showed the effects of Q_1' and $Q_1'R_1'$ to be highly significant (1%) and the effect of T_1' to be significant (5%). All these effects were positive. That is, the significant effects of the independent variables of reverse flow rate, reverse flow volume, and settling time on H_{crit} are that a linear increase in reverse flow volume will produce a linear increase in H_{crit} , that this effect is greater at the higher reverse flow rate than at the lower, and that allowing settling causes an increase in H_{crit} .

Statistical analysis of the operating gradient, m , showed Q_1' and R_1' to be highly significant (1%) and $R_1'T_1'$ to be significant (5%). The effect of Q was positive and the other effects were negative. That is, m appeared to be linearly increased by increasing Q and decreased with increasing flow rate. This last effect was more marked when settling was allowed.

Statistical analysis of $V_{\max}$ gave Q_1' as very highly significant (0.1%), T_1' and $Q_1'T_1'$ as highly significant (1%), and $Q_2'R_1'$ as significant (actually less than 2½%). All effects were positive. The significance of $Q_2'R_1'$ seemed extremely unlikely and that of $Q_1'T_1'$ seemed rather improbable. The test discussed in section 3.4 on the normality of the data raised suspicions about the settings of Q and checks on the value of $V_{\max}$ showed some to be irreproducible and led to the discovery of the errors discussed in section 2.3.1.1.

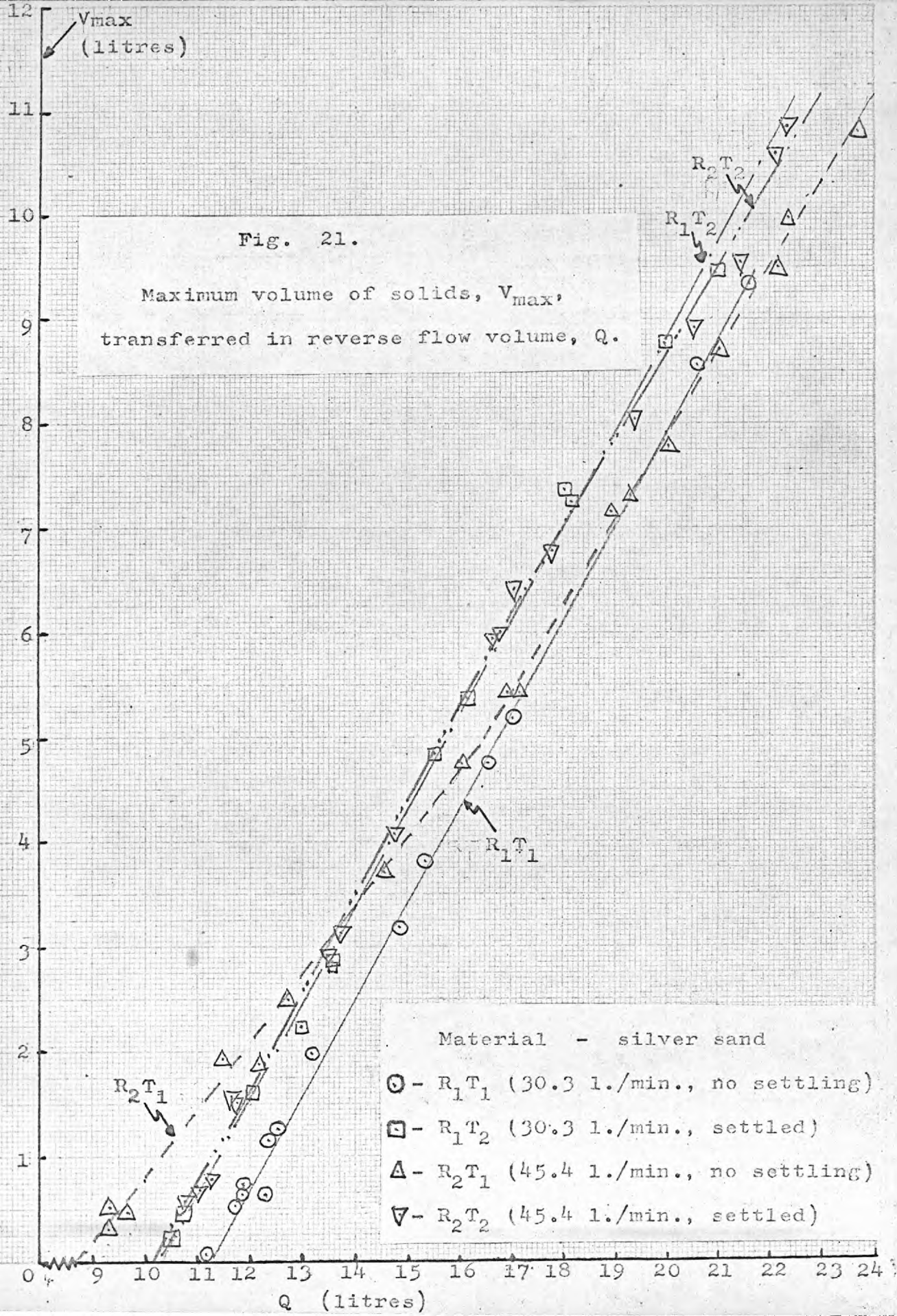
Neither H_{crit} nor m seemed particularly sensitive to small changes in Q and in the analysis of these statistics, the residual error was at least as large as estimates of experimental error. However, the residual

error in the $V_{\max}$ analysis is rather less than the anticipated experimental error.

Since it was desirable to have an accurate knowledge of $V_{\max}$, a second series of experiments were carried out with the initial stage hold-up appreciably greater than H_{crit} so that all volumes of solids transferred would be $V_{\max}$. (fig. 10). The reverse flow volume, Q , was varied and $V_{\max}$ measured at constant levels of settling time, T , and reverse flow rate, R . This was performed for both levels of settling time at each of the two levels of reverse flow rate. The results are plotted in fig. 21. The actual levels of reverse flow volume, Q , which had occurred in the factorial experiment could be read from these $V_{\max}$ versus Q plots.

The variation of $V_{\max}$ with Q appeared to be linear with the marked exception that the results at the higher reverse flow rate, R_2 , seemed to change slope at a value of Q of about 16.8 litres. Straight lines of best fit (based on least squares on the $V_{\max}$ axis) were calculated, making separate calculations for the higher reverse flow rate results above and below Q equal to 16.8 litres.

When Q was less than 16.8 litres, the outstanding feature of the plots was the relatively high level of



$V_{\max}$ at the higher reverse flow rate and without settling, R_{2T_1} . This was observably attributable to the solids and water becoming mixed in the retainer. With these conditions of reverse flow, solids passed in a jet up the centre of the retainer and left the vessel before all the water which was initially present had been displaced. In all other cases, the sand rose smoothly in the retainer. The change in slope presumably indicated the point at which water initially bypassed in the retainer had been completely expelled and the sand fraction in the retainer equalled that in the line.

The fact that the plots of $V_{\max}$ versus Q for fully settled solids, T_2 , intersected the Q axis at close to the retainer plus transfer lines volume (10.4 litres) substantiated the observation that little mixing of the dense phase solids flow and the water initially present in the retainer took place. It also indicated that there was negligible slip of water in the dense phase flow while the retainer was filling with solids.

The inflexion in the plot for the higher reverse flow rate and with full settling, R_{2T_2} , at about Q equal to 16.8 litres might indicate the expulsion from the retainer of a small volume of initially bypassed water

similar to that which was observed when there was no settling of solids, $R_2 T_1$. With fully settled solids, below Q equal to 16.8 litres, $V_{\max}$ was increased slightly with increased reverse flow velocity. This also would suggest a small amount of initial bypassing of water in the retainer.

If this interpretation were correct, it would help to explain the small interaction between reverse flow rate and reverse flow volume when the reverse flow volume was greater than 16.8 litres. This interaction could only be interpreted as due to a slight increase in slip at the higher reverse flow rate. It would be reasonable for the change in slip to occur when the retainer became filled with dense phase solids.

A small constant effect of settling time on $V_{\max}$ was shown except where the effect was masked by water being bypassed by dense phase flow in the retainer.

It was observed that at the higher reverse flow rate, R_2 , solids remained in the contactor at the end of dense phase flow to the retainer. This was a confounding effect and rendered meaningless the analysis of the retainer utilization, U , based on solids not transferred out of the stage. Hence no analysis of these

results is quoted. The values of U for silver sand are listed in Appendix 2. At the lower flow rate, R_1 , these could be used for design purposes.

3.2.2. Factorial experiment on resin.

The results of the twelve experiments of the statistical experiment on ion-exchange resin are plotted in fig. 17 to 20,

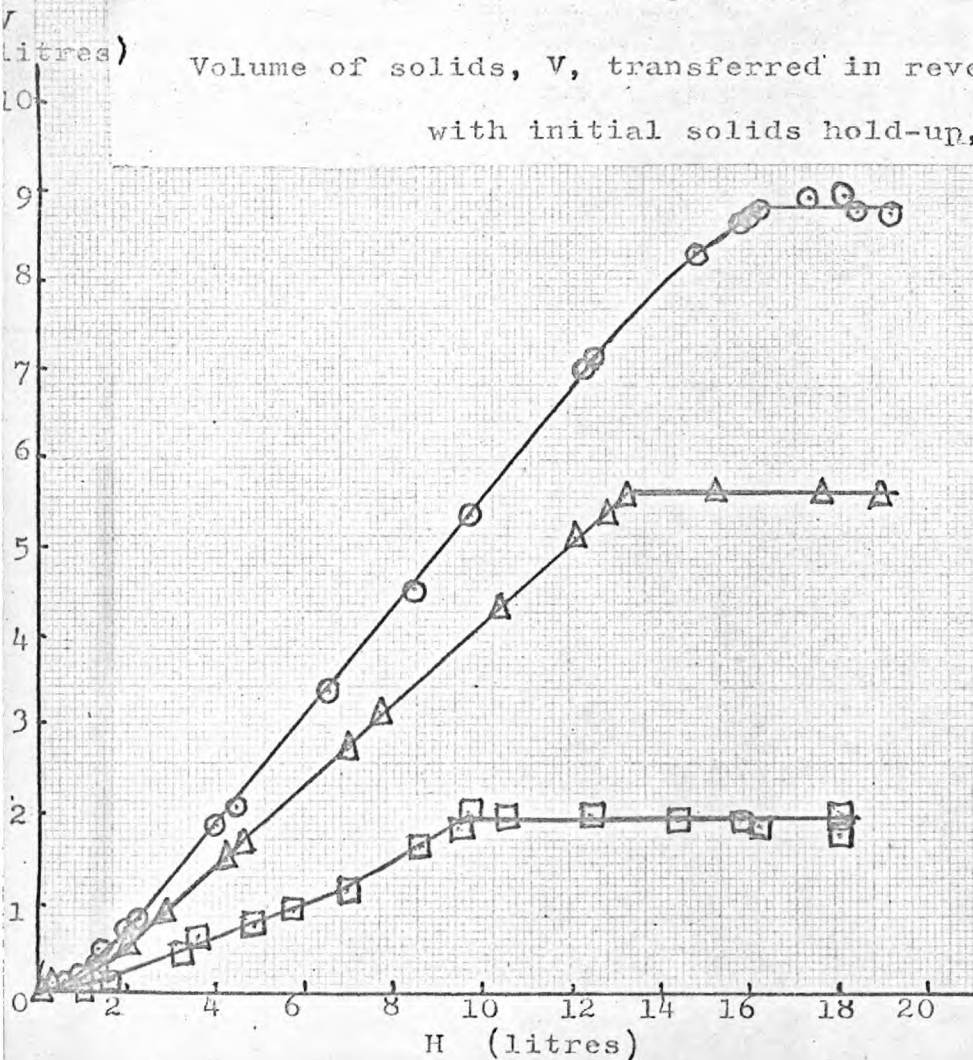
Ion-exchange resin showed no zone of low solids hold-up, H , at which no solids were transferred out of the stage in reverse flow. At the lower reverse flow rate, 30.3 litre/min., a negligible deviation towards retention of solids did occur. The effect was entirely absent at the upper flow rate, 45.4 litre/min.

The statistical analysis of H_{crit} showed the positive linear effect of Q , Q_1' , to be very highly significant (0.1%) and the negative linear effect of R , R_1' , to be significant (actually less than 2½%). Analysis of the levels of V_{max} showed the positive linear effect of Q , Q_1' , to be very highly significant (0.1%) and there were no other significant effects.

Ion-exchange resin gave a much closer approach to linear operating gradients, m , than silver sand. Analysing m indicated a very highly significant (0.1%)

Fig. 17.

Volume of solids, V , transferred in reverse flow volume, Q ,
with initial solids hold-up, H .



Material - ion ex. resin

Flow rate - 30.3 l./min.

Settling time - 0 sec.

□ - Q_1 = 12.7 litres

△ - Q_2 = 16.3 litres

○ - Q_3 = 20.0 litres

Fig. 18.

Volume of solids, V , transferred in reverse flow volume, Q ,
with initial solids hold-up, H .

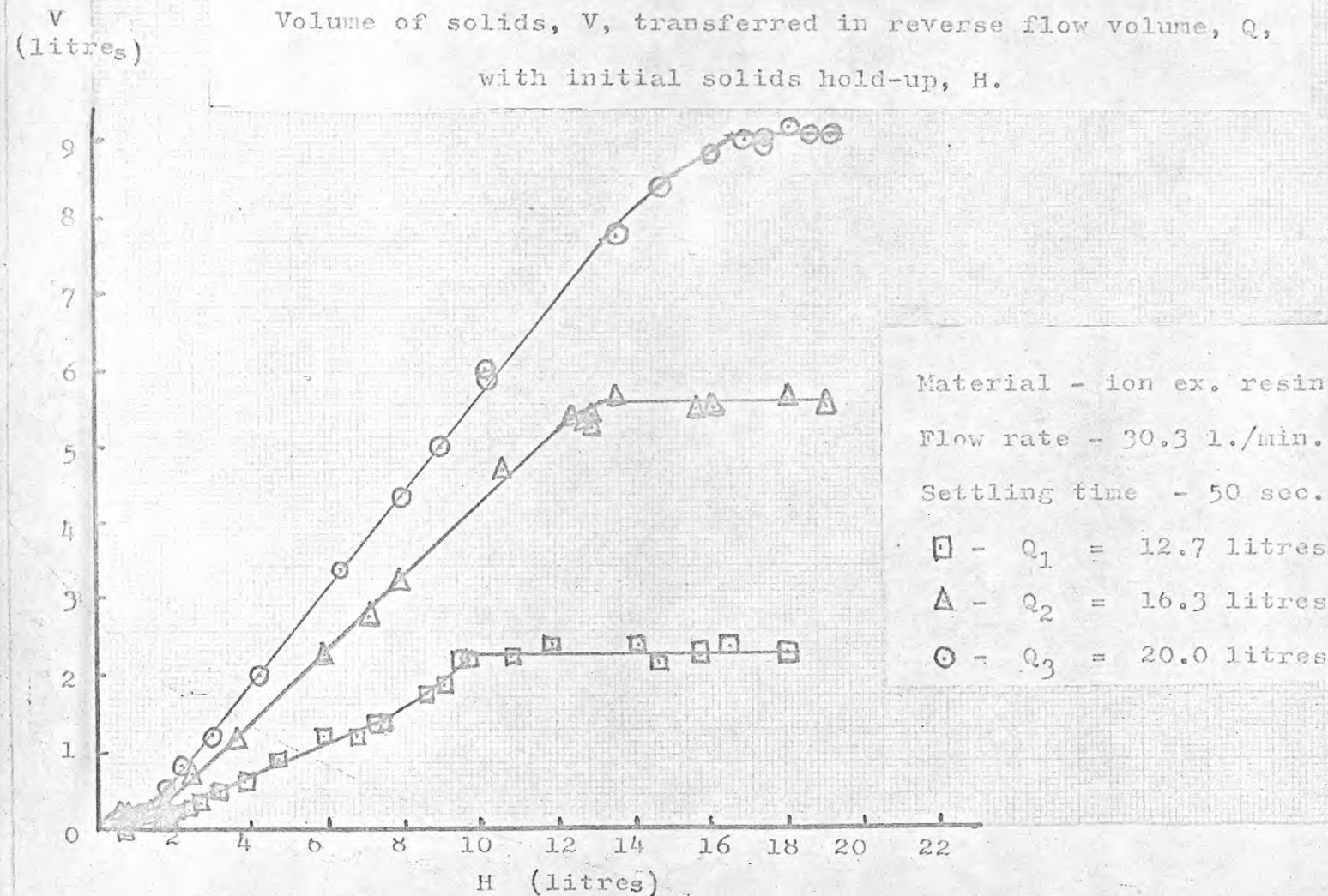
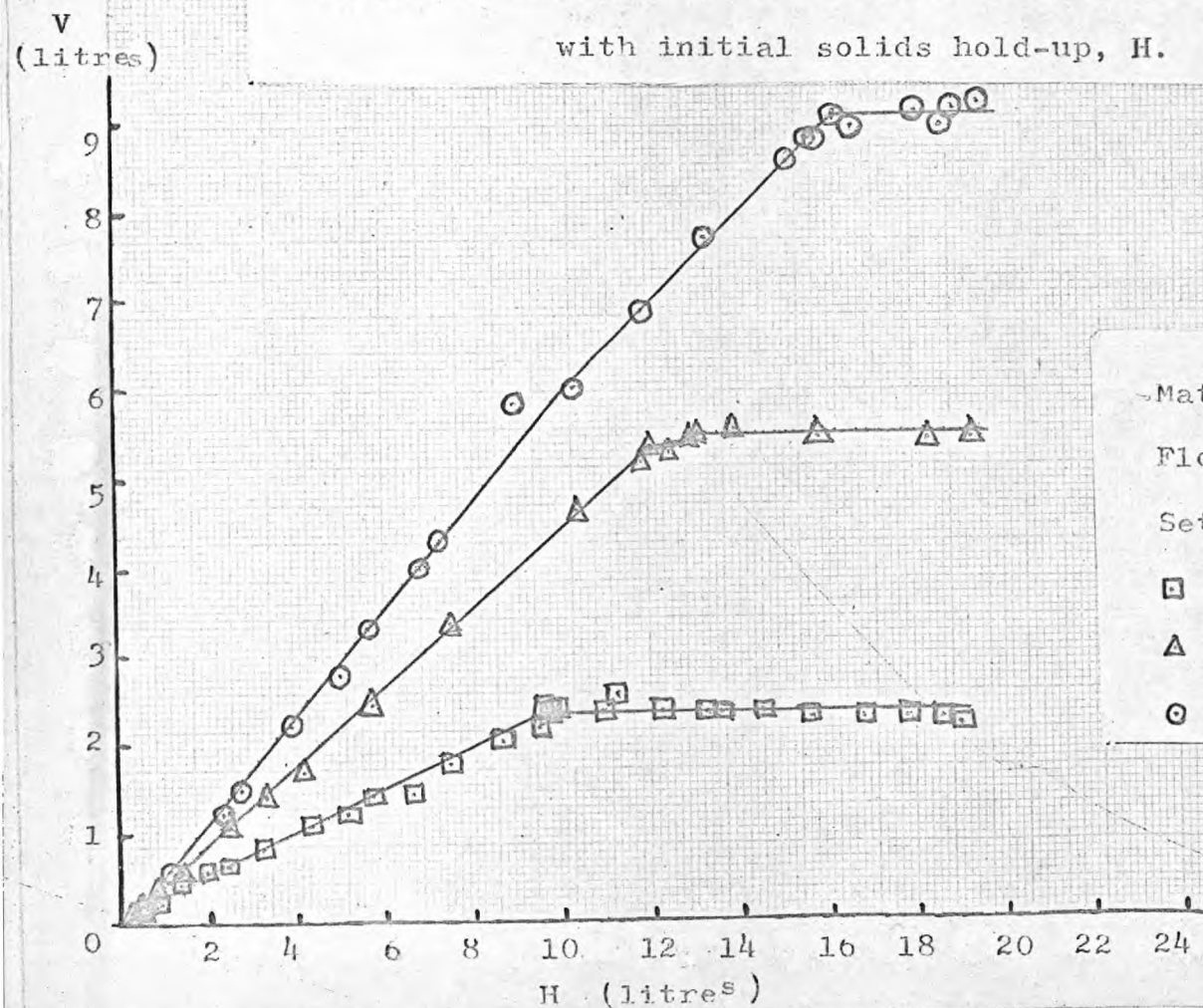


Fig. 19.

Volume of solids, V , transferred in reverse flow volume, Q ,
with initial solids hold-up, H .

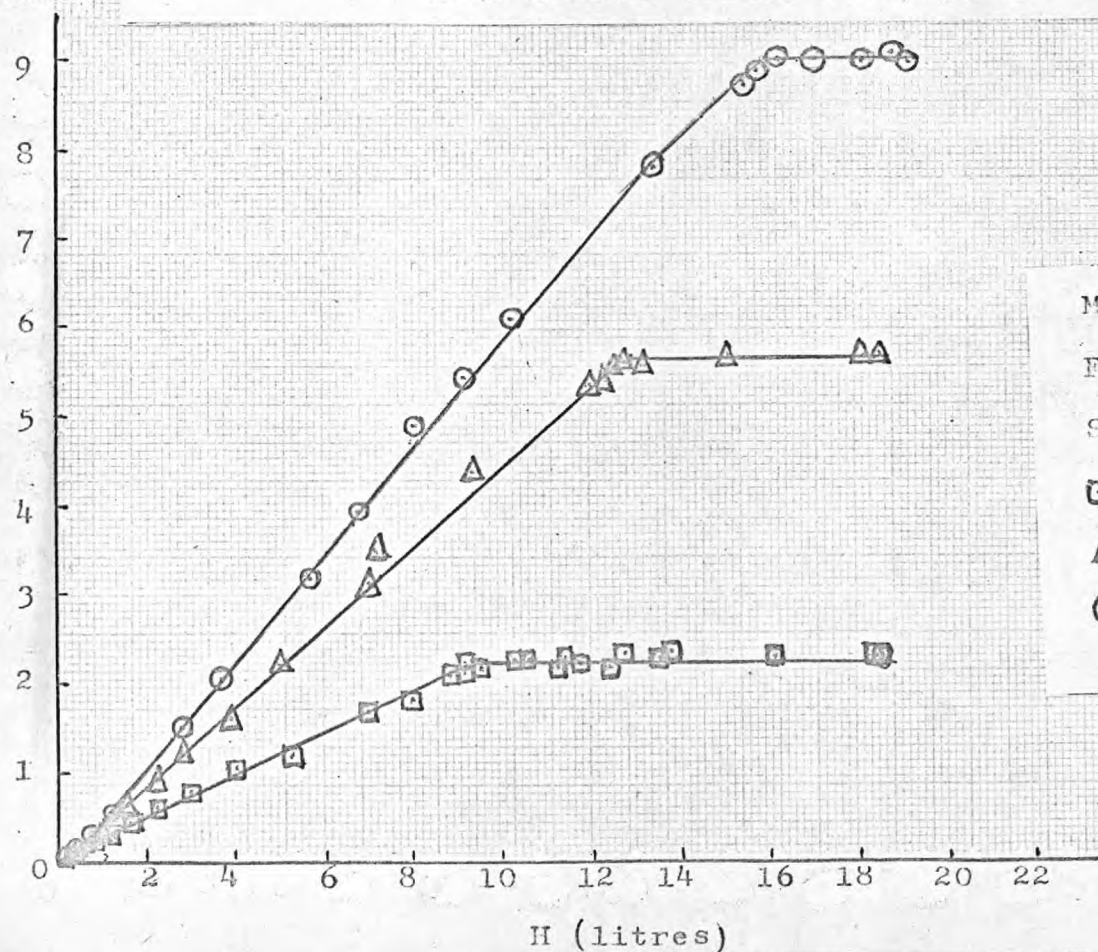


Material - ion ex. resin
Flow rate - 45.4 l./min.
Settling time - 0 sec.
 $\square$ - Q_1 = 12.7 litres
 Δ - Q_2 = 16.3 litres
 $\circ$ - Q_3 = 20.0 litres

Fig. 20.

Volume of solids, V , transferred in reverse flow volume, Q ,
with initial solids hold-up, H .

V
(litres)



Material - ion ex. resin

Flow rate - 45.4 l./min.

Settling time - 50 sec.

□ - Q_1 = 12.7 litres

△ - Q_2 = 16.3 litres

○ - Q_3 = 20.0 litres

positive effect of Q , Q_1' , and a highly significant negative effect (1%) of Q , Q_2' . Analysis of retainer utilization, U , indicated only the negative linear effect of R , R_1' , to be highly significant (1%). The values of the statistics are listed in appendix 2 with the factorial analyses.

3.3. Results expressing V as a function of Q .

The behaviour of solids in reverse flow can be summarized graphically using V versus Q co-ordinates and drawing in lines of equal hold-up as well as $V_{\max}$. These replots are shown in fig. 22 to 25 for silver sand and 26 to 29 for ion-exchange resin.

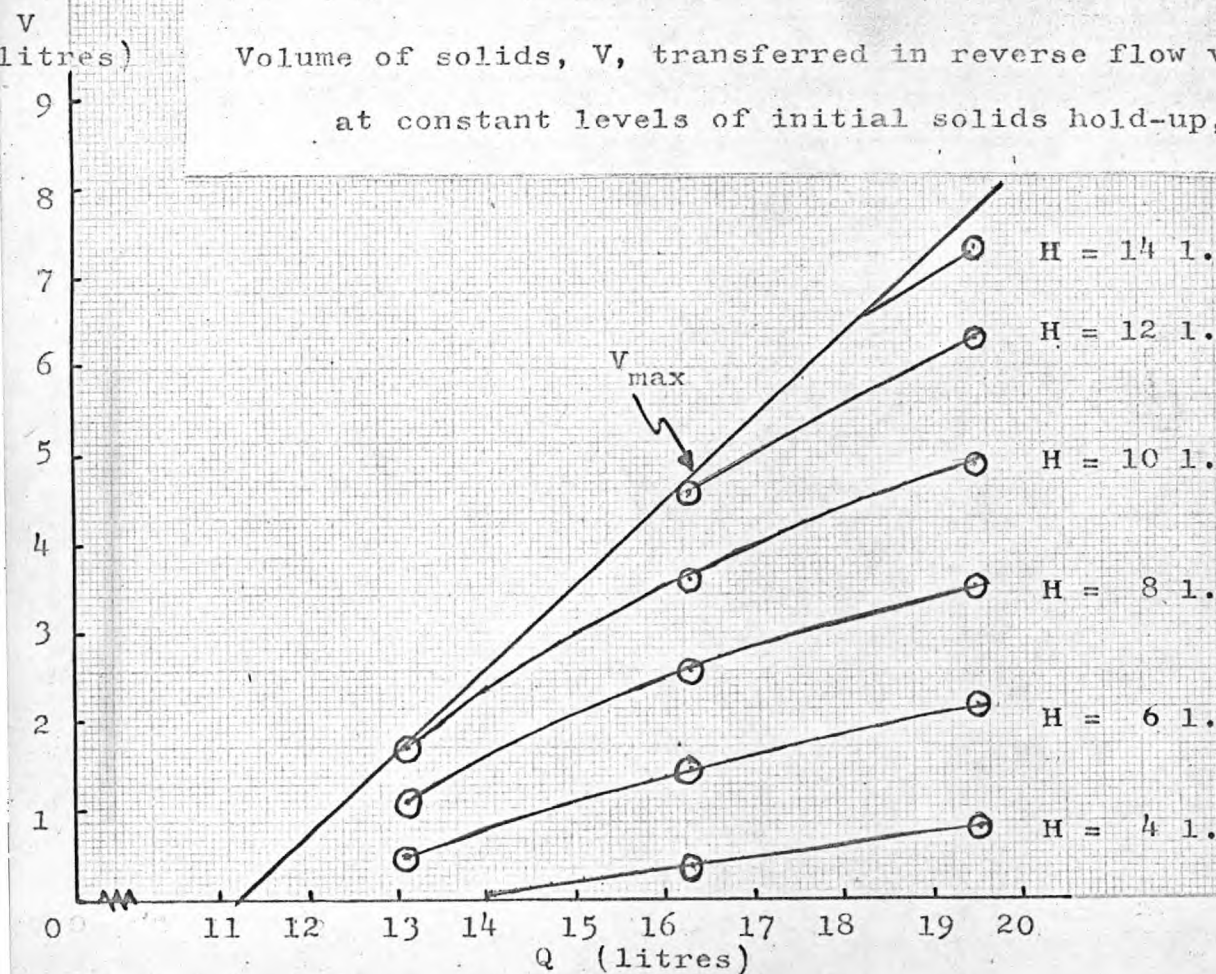
The plots for ion-exchange resin show that $V_{\max}$ varies linearly with Q . In all cases, the intersection of these plots with the volume, Q , axis is close to the volume of the retainer plus transfer lines. This indicates that there was negligible slip before the retainer was filled with dense phase solids and that very little mixing occurred in the retainer between the dense phase flow and the water which it was displacing.

3.4. Normality of data.

Bartlett tests were carried out to check the normality of the error distribution between the different

Fig. 22.

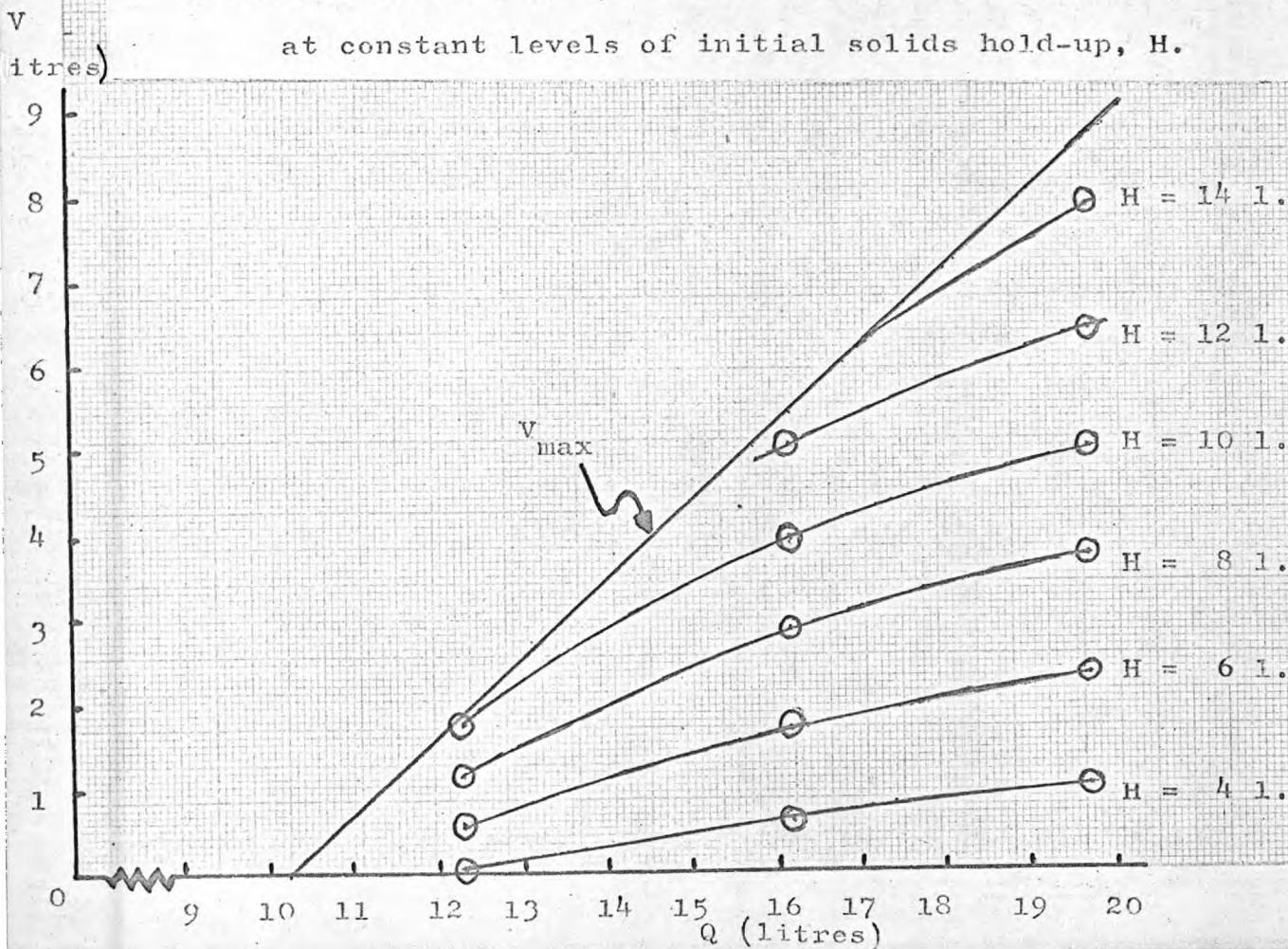
Volume of solids, V , transferred in reverse flow volume, Q ,
at constant levels of initial solids hold-up, H .



Material - silver sand
Flow rate - 30.3 l./min.
Settling time - 0 sec.
 $V_{\max}$ from fig. 21.

Fig. 23.

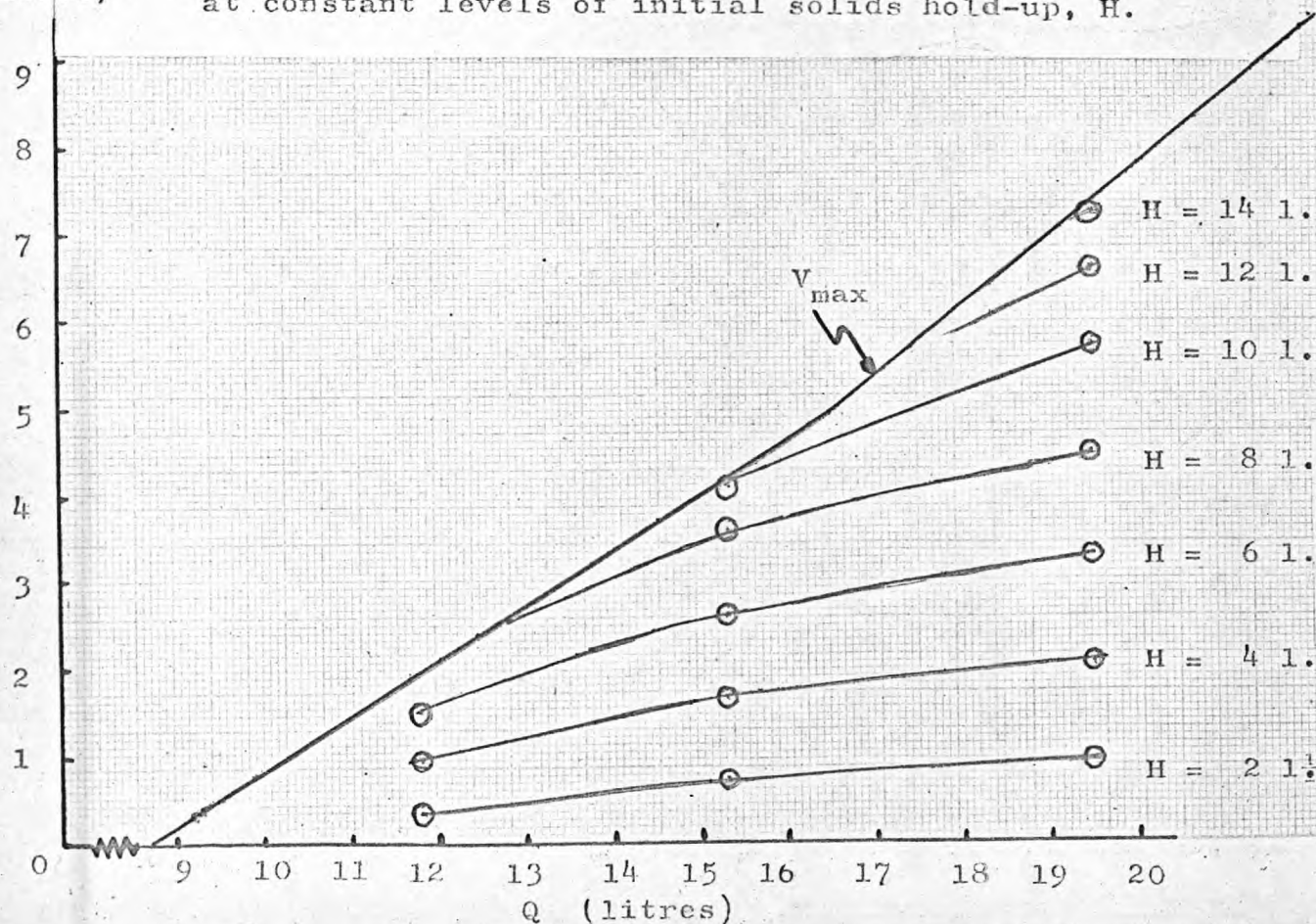
Volume of solids, V , transferred in reverse flow volume, Q ,
at constant levels of initial solids hold-up, H .



Material - silver sand
Flow rate - 30.3 l./min.
Settling time - 50 sec.
 V_{max} from fig. 21.

Fig. 24.

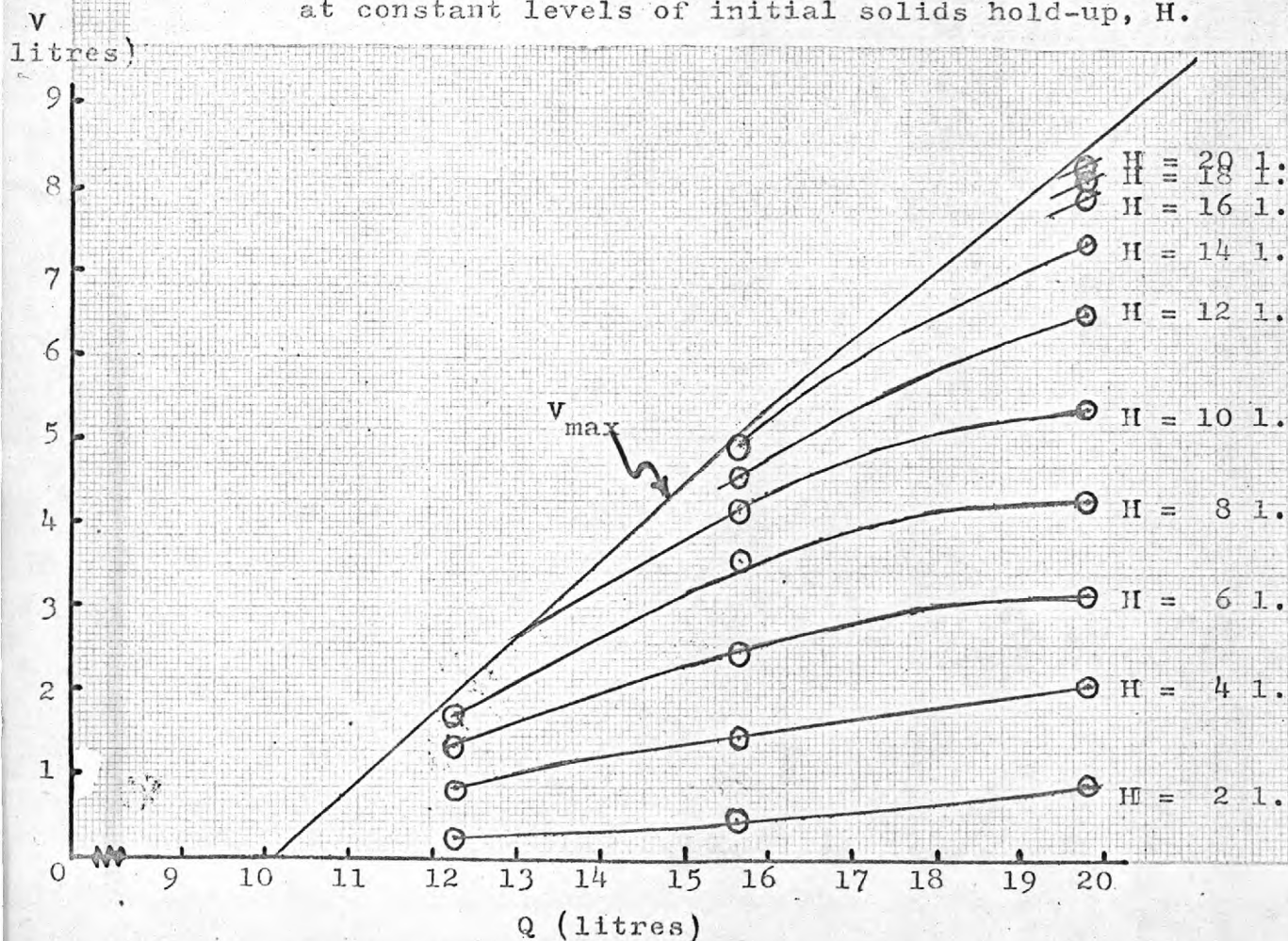
Volume of solids, V , transferred in reverse flow volume, Q ,
(litres)
at constant levels of initial solids hold-up, H .



Material - silver sand
Flow rate - 60.6 l./min
Settling time - 0 sec
 $V_{\max}$ from fig. 21.

Fig. 25.

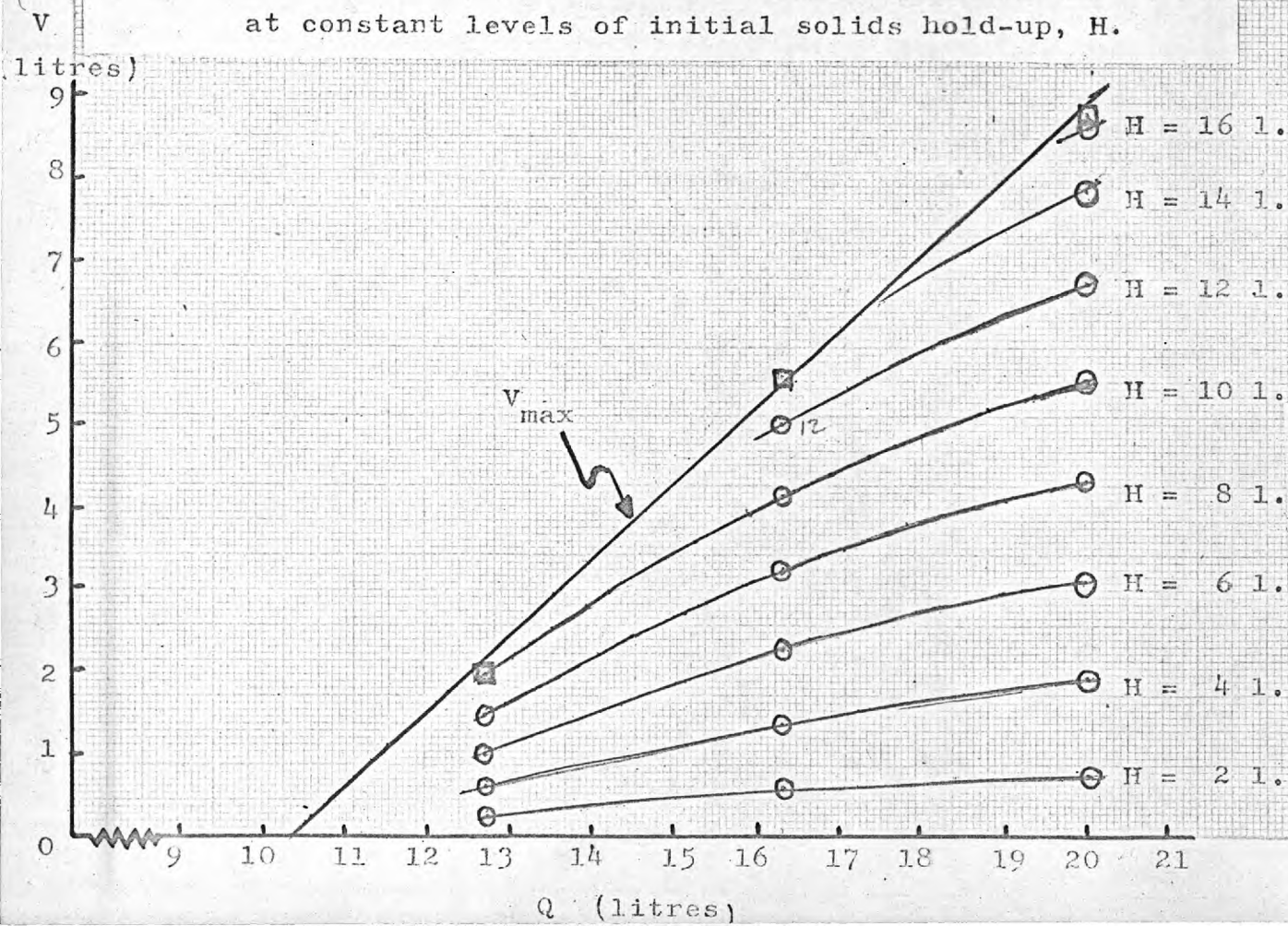
Volume of solids, V , transferred in reverse flow volume, Q ,
at constant levels of initial solids hold-up, H .



Material - silver sand
Flow rate - 60.6 l./min.
Settling time - 50 sec.
 $V_{\max}$ from fig. 21.

Fig. 26.

Volume of solids, V , transferred in reverse flow volume, Q ,
at constant levels of initial solids hold-up, H .

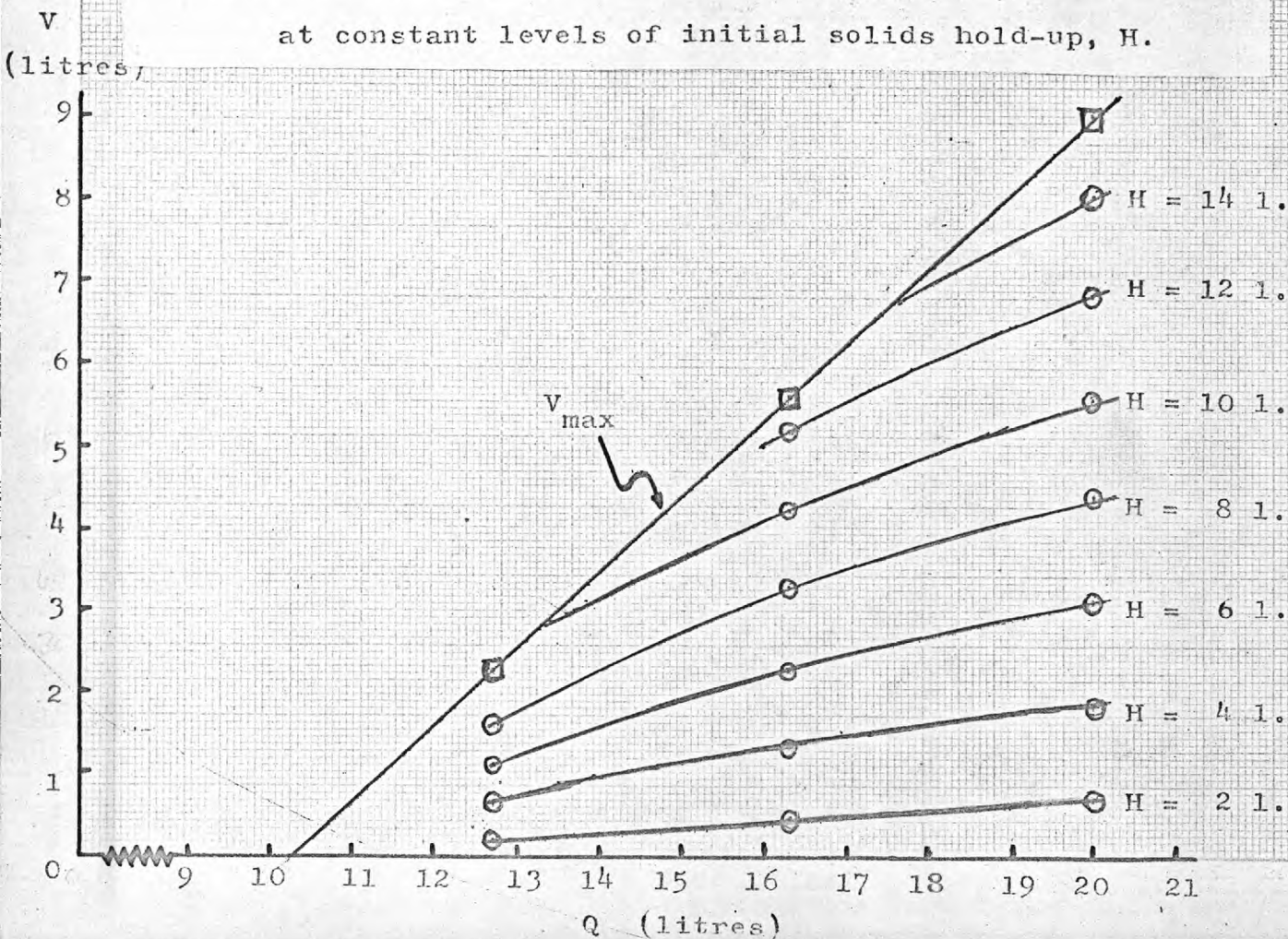


Material - ion ex. resin
Flow rate - 30.3 l./min
Settling time - 0 sec

□ - V_{max}

Fig. 27.

Volume of solids, V , transferred in reverse flow volume, Q ,
at constant levels of initial solids hold-up, H .



Material - ion ex. resin
Flow rate - 30.3 l./min
Settling time - 50 sec

$\square$ - V_{max}

Fig. 28.

Volume of solids, V , transferred in reverse flow volume, Q ,
at constant levels of initial solids hold-up, H .

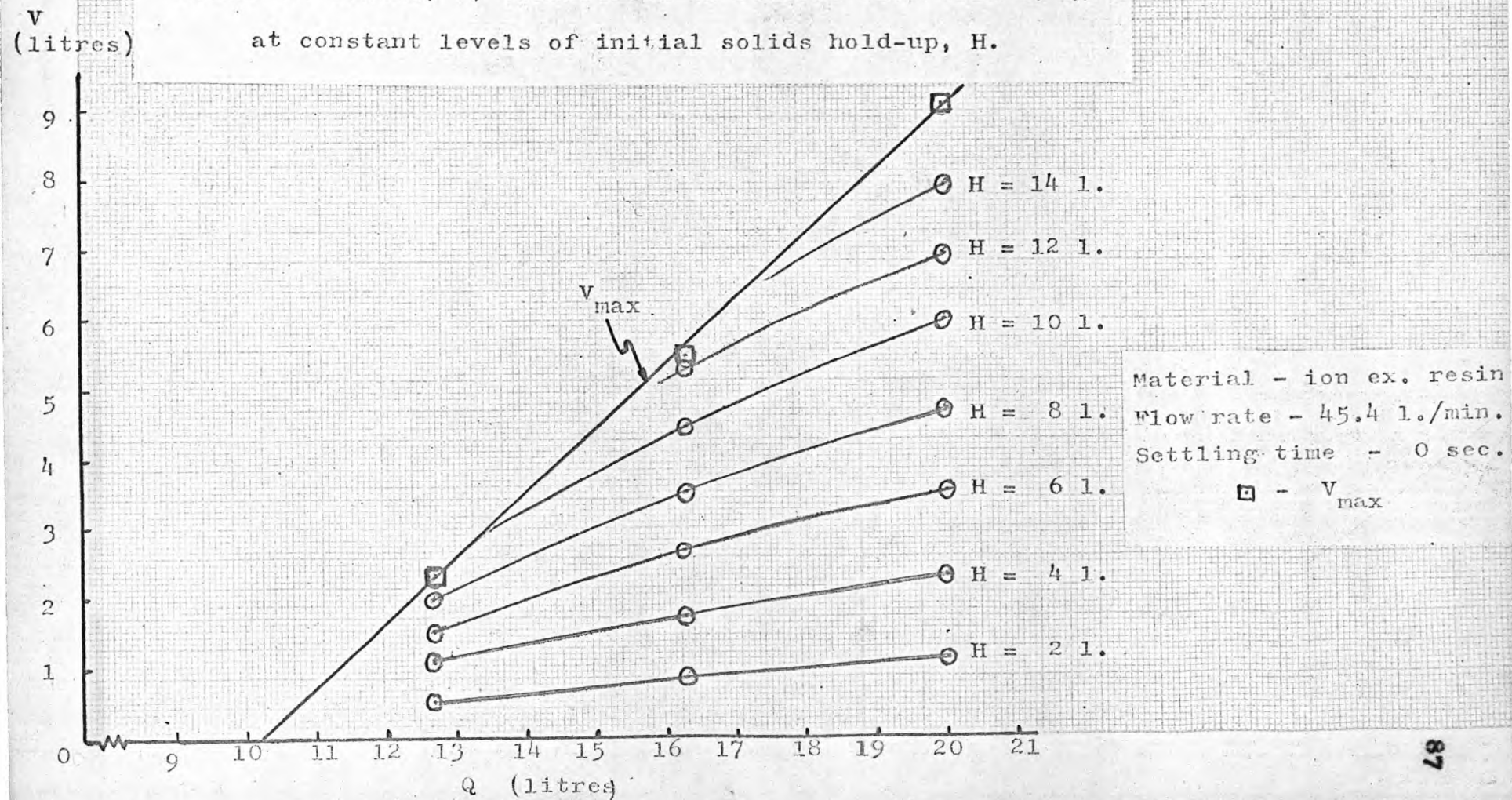
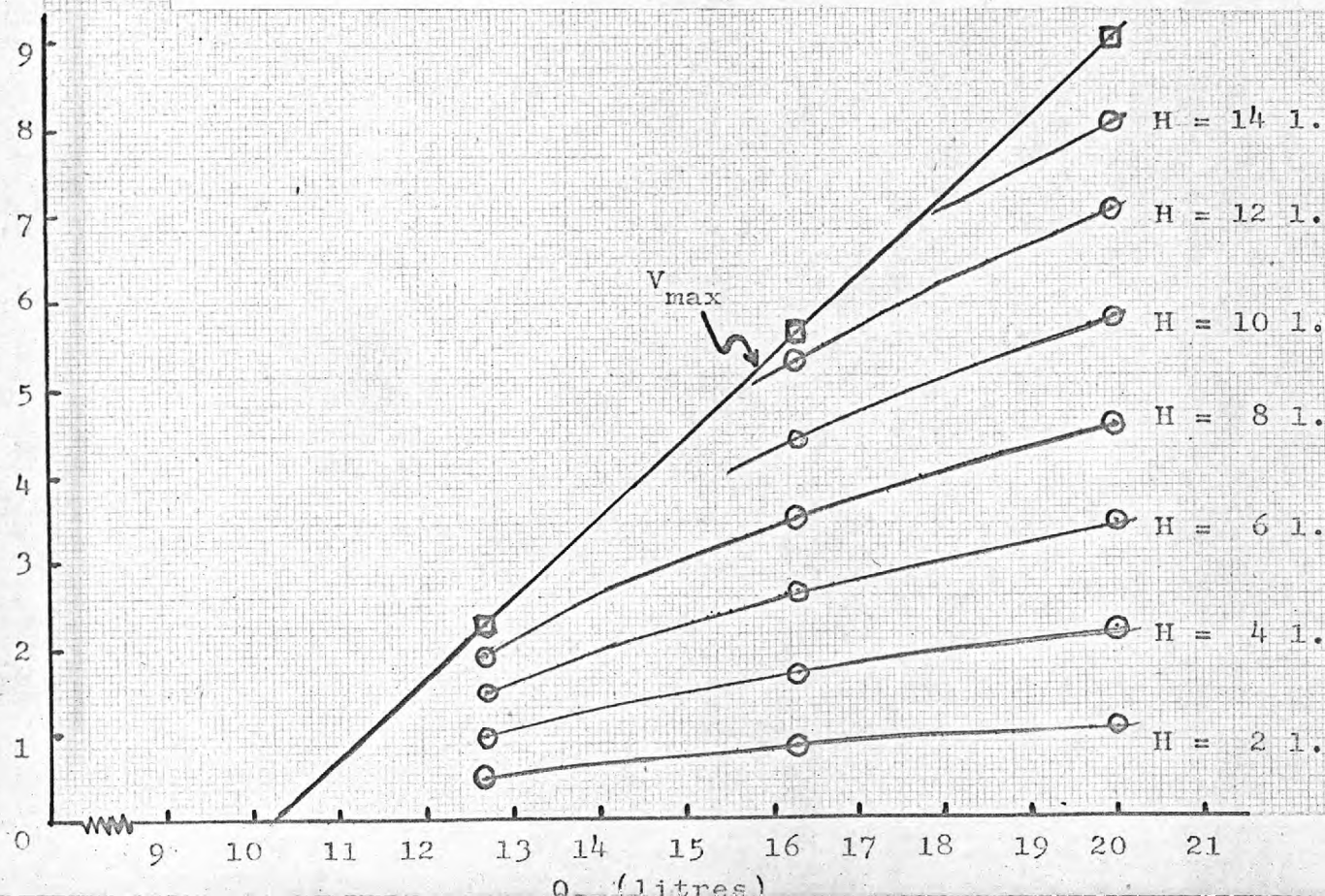


Fig. 29.

Volume of solids, V , transferred in reverse flow volume, Q ,
at constant levels of initial solids hold-up, H .

V
(litres)



Material - ion ex. resin

Flow rate - 45.4 l./min

Settling time - 50 sec.

□ - V_{max}

levels of Q . None of the six tests - of $V_{\max}$, H_{crit} , and m for silver sand and for resin - were significant. However, in the case of $V_{\max}$ for the silver sand the probability of the error distribution being random fell to almost 10% which suggested the tendencies of non-normal error distribution associated with setting Q discussed above. By comparison, Bartlett tests on the normality of errors in the two populations of $V_{\max}$ at the two levels of settling time and of reverse flow rate gave 95 and 99.5% probabilities that the departures of the error distributions from normality were purely random. The corresponding test of normality of error between different levels of Q populations of $V_{\max}$ for the ion-exchange resin gave a greater than 70% probability of normal distribution of error.

Hence, with one exception, the error distributions for the various statistics appear to be normal and the results of the factorial analysis can be accepted with confidence. The exception is $V_{\max}$ for silver sand and no conclusions have been drawn from the factorial analysis of this statistic.

3.5. Slip ratio.

Slip ratio is defined throughout these experiments

as the absolute liquid velocity past the solids expressed as a percentage of the solids velocity. That is:

$$\begin{aligned}
 \text{Slip ratio} &= \frac{(\text{linear velocity, liquid} - \text{linear velocity, solids})100}{\text{linear velocity, solids}} \% \\
 &= \frac{100 \left[\frac{Q_s \cdot e_d}{A t \cdot e_t} - \frac{Q_s(1 - e_d)}{A t(1 - e_t)} \right]}{\frac{Q_s(1 - e_d)}{A t(1 - e_t)}} \% \\
 &= \frac{100(e_d - e_t)}{e_t(1 - e_d)} \%
 \end{aligned}$$

where Q_s = total volume of solids and liquid delivered
in time, t ,

A = cross-sectional area of the pipe,

e_t = true voidage in the line,

e_d = fraction of water in Q_s .

The delivered voidage, e_d , could easily be calculated since the volume of solids conveyed in a reverse flow volume was measured. This was usually expressed as a free-settled bulk volume but could be converted to an absolute volume since this was a constant fraction of the free-settled bulk volume for any one

material. Q_s was taken to be the total reverse flow volume, Q , less the volume of the retainer plus transfer lines since only solids leaving this section were measured. Slip before this section was filled with solids could be ignored since it has been shown to be negligible. (Sections 3.2.1. and 3.3)

The true voidage, e_t , was measured in the retainer by stopping reverse flow suddenly when the retainer was full of dense phase flow. The free-settled volume could then be measured from the height of the solids in the retainer using a previous calibration of the volume of the upper reducing section of the retainer.

Slip ratios of between 17 and 22% were noted for silver sand and of between 15 and 17% for the ion exchange resin. These values would provide only an approximate indication of the slip ratios since they were based on the voidage of solids in the retainer which is not necessarily an accurate measurement of voidage in the 1 inch link lines.

4. DISCUSSION AND CONCLUSIONS.

4.1. Discussion of the analysis of the results.

The less complex results of the analyses for ion-exchange resin reveal a very simple situation. In fact, no effects were detected which could not have been foreseen before the experiments had been performed. The absence of certain other effects which might also have been anticipated is at least as useful a result of the experiments. This is one of the advantages of factorial experimentation whereby effects can be shown to be insignificant when compared with the residual mean squares estimate of error.

The only significant effect on retainer utilization is that of reverse flow rate. To understand the mechanism of retainer utilization, consider the hypothetical case of an infinitesimal reverse flow rate. When reverse flow has emptied the contactor and the transfer line to the retainer of solids, water will enter the bottom of the retainer. At the moment when water enters the retainer, this vessel is filled with dense phase solids. Since the flow rate is infinitesimal, no dense phase solids will leave the retainer while the water is penetrating upward from the bottom to the top of

the retainer. By definition, retainer utilization is determined at the point when dense phase flow out of the retainer is ending. Hence, with an infinitesimal flow rate, retainer utilization will be one.

If the reverse flow rate is increased, some dense phase solids will leave the retainer before water - or dilute phase flow in actual conditions - penetrates through the dense phase solids and the retainer utilization will be less than one. The rate of upward penetration of dense phase flow would be expected to be relatively constant referred to the frame of reference of the moving dense phase solids. Hence, the quantity of solids displaced from the retainer before the penetration of dilute phase flow to the top of the retainer will be increased by reverse flow rate. This is the effect shown.

The volume of dense phase which has passed out of the stage prior to the entry of dilute phase flow to the retainer would not be expected to affect retainer utilization. Hence, there is no effect of reverse flow volume, Q . Similarly, if, in the absence of settling, some dense phase flow was replaced by water at the beginning of reverse flow, this water would have been expelled from the retainer before dilute phase flow entered this vessel

unless the reverse flow volume was very small. Therefore, no effects of settling time would be expected from the analysis of retainer utilization.

H_{crit} is also reduced by increasing reverse flow rate since there is a smaller solids volume in the retainer at the end of reverse flow and since analysis of V_{max} shows that the volume transferred out of the stage is not significantly different at the two reverse flow rates. The linear increases in V_{max} and H_{crit} with increasing reverse flow volume, Q , would be expected.

The two effects of reverse flow volume on the operating gradient, m , would be expected. However, the absence of any other effects is of interest. Apparently, the retainer belied its name and there was completely uniform mixing of solids and liquid in the retainer. If this had not been so, an effect of reverse flow rate would have been expected since any tendency of solids to settle would have been influenced by the reverse flow rate.

The absence of any effects of settling time, T , on any of the statistics is surprising and its significance is discussed below.

The effects on the statistics for silver sand are more complicated.

The effects on $V_{\max}$ are interpreted in section 3.2.1. However, it is worth noting that the predominant effects of Q and T appear in the analysis of the factorial experiment despite the errors in setting the reverse flow volume, Q . The apparently spurious significant effects indicated by this analysis are all less significant than the effects of Q and T .

H_{crit} shows the expected effect of the reverse flow volume, Q . The effect on H_{crit} of settling time, T , is easily explained since water in an expanded fluidized bed could replace sand in the volume of sand and water passing through the retainer at the start of reverse flow. The interaction of reverse flow rate, R , and reverse flow volume, Q , is caused by the water bypassing sand in the contactor at the higher reverse flow rate. This interaction indicates that the bypassing was greater with larger reverse flow volumes.

The operating gradient, m , is influenced by Q in the same predictable way that was noted for ion-exchange resin. However, the effect of reverse flow rate, R , only appears with silver sand and indicates that there was some

preferential retention of solids in the retainer at the lower flow rate. The interaction of R and T can be interpreted as an effect of the shorter mixing time in the retainer of small quantities of hold-up when the solids enter the retainer in dense phase flow. Without settling and the small hold-ups of less than H_{crit} , most of the solids flow to the retainer would be in dilute phase. The importance of the duration of dilute phase flow through the retainer on the volume of solids retained has already been noted in the reduction, as Q is increased, of the level of hold-up giving a V_{max} of zero.

All these effects on H_{crit} and m are fairly definite and seem unlikely to have arisen from non-normality of error distribution.

The most marked difference between the sand - water and the resin - water systems is the complete absence of any effects associated with settling time, T, with the ion-exchange resin. This is particularly surprising when the fluidization characteristics of the two solids (Appendix 1) are examined. While the characteristics are similar, the increase in voidage with increasing flow rate is rather more rapid and begins at a

slightly lower velocity. . with the ion-exchange resin.

Both solids were fully settled at the end of the settling period allowed, so the difference in the effect of settling time must arise from a difference in the fluidized beds which would appear when no settling was allowed. If the fluidization was evenly distributed, the plots of voidage against fluidizing velocity indicate that the resin would show at least as large an increase in void volume as would the silver sand. However, the absence of an effect of settling time with resin implies that there is a much smaller increase in voidage with this material than with the silver sand. Such a difference would be particularly important close to the apex of the cone since solids in the upper parts of the bed have some time to settle between the start of reverse flow and their leaving the contactor.

There is one large difference between the two solids. The silver sand has an apparent density in water more than five times as large as that of ion-exchange resin. Hence, the force exerted by water flow on unit volume of silver sand to support it against gravity is very much greater than the force on the same volume of resin. With sand, interaction between water flow and

solids would be likely to result in much greater retardation and deflection of the water flow than would occur with resin. Hence, it seems probable that with resin, there is little damping of water flow and that a high velocity jet of water can penetrate far into the bed. The water in such a jet would have a relatively short residence time and the increase in voidage in the bed - at least close to the apex - would be slight.

On the other hand, with silver sand, the jet of water into the base of the solids bed would be expected to be rapidly slowed and diffused across a much wider cross-section of the bed with increased residence time and voidage.

Studies of spouted beds by Mathur and Gishler (50) indicate that a bed of solids of high density requires much higher fluid velocities to cause spouting than would be required for a bed of low density solids but which was otherwise similar.

The other marked difference between the sand - water and the resin - water systems is in the performance of the retainer.

With resin the linear relationship of the volume of solids transferred, V , to the initial solids hold-up, H , shows that mixing is complete in the retainer

and that the volume of solids transferred out of the stage depends only on the volume present in the retainer.

With sand, retainer performance is more complicated. The phenomenon is best expressed as a delay in lifting solids through the height of the retainer. Where there is no solids transfer from the stage, the maximum level of solids hold-up is certainly dependent on the reverse flow volume of water, Q , and, at these low levels of solids hold-up, the much expanded fluidized bed in the retainer was observed to have an upper surface which rose steadily during reverse flow. This last phenomenon was not observed with ion-exchange resin. The interaction on operating gradient between settling time and reverse flow rate, discussed above, also suggests a mechanism of solids transfer through the retainer which is time dependent.

It may be that a high velocity jet of water entering the base of the retainer is less slowed by the resin than by the sand. The higher velocity jet might be more effective in lifting solids to the top of the bed.

However, no data are available on the free-settling velocities of the two solids and this is a characteristic of solids which is very difficult to

compare unless the solids particles are uniform in shape and size. All that can be said about settling velocities for the two materials is that they appear qualitatively to be not widely different. In the absence of an effective comparison of settling velocities, the explanation based on jet effects must be treated with scepticism.

4.2. Discussion of the feasibility and operation of the multistage technique.

The wider significance of the results is to the operation of a solids-liquid multistage fluidized countercurrent contactor.

The most important aspect of the results is the existence of the plateau section in the V versus H plot in every case of the two factorial experiments. This region in which the solids hold-up has no effect on the volume of solids transferred by a given reverse flow volume is vital to establishing the stability of a multistage system. If the volume transferred had shown any decrease in the plateau section with increasing reverse flow volume, a disturbance in the stage hold-up would have started a continuous upwards divergence from the desired stage solids inventory.

Operating with an H_{crit} hold-up in all stages, a multistage system composed of identical stages would

be stable. However, there is no mechanism to inhibit a drift towards increasing hold-up. In practice, this would almost certainly arise through even the smallest variation in the volumes of the retainers plus transfer lines of the individual stages.

On the other hand, the system is completely stable to small decreases in stage hold-up from H_{crit} since this diminishes subsequent solids transfer from that stage. By reducing the input of solids per cycle to the apparatus to rather less than V_{max} , the stages would operate with a hold-up of slightly less than H_{crit} and a small quantity of dilute phase solids reverse flow out of the stage would occur at the end of the reverse flow period.

The maximum possible throughput of solids of the multistage contactor is the throughput of solids of the stage with the largest retainer plus lines volume.

The volume of reverse flow, Q , per cycle can be determined from the relationship:

$$Q = A + V_{max} \left(1 + \frac{S \cdot e t}{100}\right) + c \dots \dots \dots (1)$$

where Q = volume of the reverse flow,

A = volume of the retainer + transfer lines,

$V_{\max}$ = maximum possible volume of free-settled solids
transferred in reverse flow from the stage,

S = slip ratio,

e_t = true voidage in the line,

c = a small constant when no settling of
solids is allowed.

This equation follows from the plots of $V_{\max}$ versus Q and is strictly applicable for design purposes to stages operating with a hold-up of H_{crit} . However, it is assumed that, in practice, reduction in solids hold-up from H_{crit} will be small. Equation 1 is not valid in the high reverse flow rate with no settling case of silver sand at low values of reverse flow volume.

In practice, it has been shown that $S \cdot e_t / 100$ and c are relatively small and

$$Q = A + V_{\max}$$

could be used as an acceptable approximation.

The other equation for use in designing a multi-stage system is:

$$H_{crit} = V_{max} + UA \dots\dots\dots (2)$$

where U = retainer utilization.

The size of H and V can only be decided from a knowledge of the conditions of mass transfer of a potential process and the bed hold-up which will give the optimum reaction for the amount of material involved at the selected forward flow rate. An upper limit on V would be determined by the acceptable fluctuation in the liquid product. For the reasons discussed below, there would be a preference for making V as large as possible.

With resin, and probably with materials of similar density of only slightly greater than the liquid medium, $m \propto V/H$. Although the relationship between V, H, and m is more complex with the denser silver sand, the same trend is followed.

The plots of V versus H confirm that a stage filled only with liquid can be filled with solids merely by adding solids to the first stage (relative to solids movement through the system) in the same way as in normal operation. By adding the normal volume of solids for the particular reverse flow conditions, the volume of solids in all stages will steadily build up to the operating hold-up

volume. No special start-up procedure is required, but the speed of this build-up is controlled by m , the operating gradient and a high m also gives faster return to the normal solids stage inventory in the event of any disturbance of solids feed.

For example, consider three three-stage contactors filled with liquid with operating gradients of 0.75, 0.5 and 0.25. After a volume of solids equivalent to three equilibrium stage hold-ups has been introduced in the appropriate number of reverse flow periods (4, 6 and 12 respectively), the contactors will have solids hold-ups of 89.5, 84.4 and 79.0 percent of the equilibrium hold-up, respectively.

This effect on the rate of build-up of solids is not markedly significant.

The $V:H$ ratio (which is not always exactly m) gives a more significant effect. The solids hold-up will normally be completely transferred into the retainer in a reverse flow period. When V is small relative to H (that is a low m) a relatively great amount of liquid is transferred in reverse flow with the transfer of a small volume of solids. Reverse flow must be repeated several times to transfer a stage hold-up of solids, H , between adjacent stages.

In contrast, with high values of m there is relatively less liquid associated with the transfer of solids in each reverse flow period and the number of reverse flow periods to transfer a stage hold-up between adjacent stages is reduced. Clearly, higher solids and liquid throughputs are possible with high values of m than with low values.

For this reason it would be desirable to operate with the highest ratio of V to H which produces neither unacceptable fluctuations in the concentration of liquid product or substantial solids transfer through two stages in one reverse flow period.

Having selected values for H and V , the volume of the retainer is given by equation (2) and the size of the reverse flow volume, Q , by equation (1). In sizing the contactor an allowance of 100% expansion to the settled bed volume would probably be about normal. Assuming a fairly typical retainer utilization of 60% and a reverse flow transfer of solids of half the solids in the stage per flow cycle ($V/H = 0.5$), the contactor would be 2.8 times the size of the retainer. If $V/H = 0.25$, the contactor to retainer ratio becomes 1.9:1 but for most applications a ratio between contactor and retainer of not less than 2.5:1 would be about the

minimum to give any flexibility of operation and 3:1 would probably be advantageous.

It has been shown in Part II of this study that there is a theoretical lower limit to the reverse flow rate below which point there is insufficient slip of water past solids to fluidize the solids in the retainer. This produces a packed bed in both the retainer and the transfer lines.

Operating with reverse flow rates close to this lower limit would be marginally advantageous with denser solids materials such as silver sand since it gives a higher operating gradient, m , with the associated increase in rates of build-up of solids hold-up.

Allowance would have to be made to provide enough pumping power to clear solids from the transfer lines and the retainer in the event of an accidental incursion below the minimum velocity while the retainer was packed with solids. This power requirement is easily calculated from the published data (33) on pressure drop through fixed beds since flow is not resumed until the slip rate through the line linking the bases of the contactor and the retainer is sufficient to fluidize the solids in the retainer. Until this occurs, the solids in the linking line are themselves in a packed

non-fluidized state. Solids in the linking line between the top of the retainer and the contactor are not in any way restrained and are fluidized with much lower slip rates. Hence the pressure required to clear a blockage could be minimised by making the link between the bases of the contactor and of the retainer as short as possible. Too wide lines would be undesirable since they would interfere with the performance of the retainer and solids would tend to be held in them on the resumption of fluidizing forward flow.

4.3. Conclusions.

These experiments indicate that a multistage solids-liquid contactor of this type and of the pilot plant size studied could maintain a stable solids inventory in each stage. It appears that solids hold-up would be completely stable at any value up to the critical hold-up. In practice, since it is preferable to transfer solids in reverse flow with the minimum of the countercurrent liquid phase, it would be advisable to operate with a transferred solids volume, V , as close to the maximum transferred solids volume, $V_{\max}$, as the variation in retainer volume between stages would allow.

Use of the largest ratio of volume of solids transferred to volume of solids in a stage which is consistent with acceptable fluctuation in the liquid output and avoiding transfer of solids through two stages in a single reverse flow period is recommended.

Use of the lowest possible reverse flow rate appears to be marginally preferable. A lower limit on the reverse flow rate has been shown to exist and the experiments of part II show that this corresponds to minimum fluidizing conditions in the retainer.

There would appear to be a small advantage in

allowing a settling period in the operating flow cycle of the technique for a dense material such as silver sand and the significance of allowing settling would be greater with higher forward flow rates.

A method is indicated for the sizing of vessels and reverse flow volume if the solids hold-up in each stage and the volume of solids to be transferred per cycle has been determined.

PART II.

STUDIES OF VERTICALLY UPWARD CONVEYING OF SOLIDS
IN WATER IN DENSE PHASE FLOW.

5. INTRODUCTION.

5.1. General introduction.

In the contactor studied in part I, solids move between the stages in the contactor in "dense phase flow". This state of solids transport arises because the solids are not pumped directly but move with the assistance of gravity from a settled bed of solids into a pipeline. The solids enter the pipe in virtually settled packing.

In order to move the solids against the resisting forces, the interstitial water has to travel faster than the solids. That is, there is a slip velocity of the water past the solids. When the direction of flow changes from the initial vertically downward to vertically upward, the interstitial water has to do more work in moving the solids against gravity and the slip velocity must increase. To accommodate increased velocity, the cross-sectional area occupied by water must decrease and hence the voidage becomes less than in the free-settled state.

Throughout the investigations of dense phase flow, the solids fraction in the line was always greater than that of free-settled solids. Thus, the dense phase flow of this study can be arbitrarily defined as the flow of solids with voidage less than that occurring in the free-settled state.

Beside being an inherent feature of the flow of solids in the contactor described above, dense phase flow might be applied to pipeline transportation. Its use would give a marked reduction in the quantity of accompanying liquid from that occurring with the dilute phase flow used in conventional hydrodynamic conveying. For example, dense phase flow could be used with advantage in adding and withdrawing solids with a continuous solids-liquid multistage contactor.

A literature search did not reveal any work on solids-liquid conveying with solids concentrations as high as those of dense phase flow, except at very low velocity. Hence, it was decided to make a closer study of dense phase flow and to compare it with dilute phase flow.

5.2. The present investigation.

Since solids transference in the solids-liquid contactor described earlier involved predominantly vertical lift operations, it was decided to concentrate the study on the upward flow of solids. To do this adequately required measurement of the pressure drop across a straight section of vertically upward flow, the concentration of solids flowing vertically upward in the line, the delivered concentration of solids, and the flow velocity.

Empirical measurements of the pressure drops over more complex sections of the piping could also be made but it was not practical to extend the study to horizontal dense phase flow without much elaboration. Difficulty would arise in horizontal flow due to measurement of the concentration of flowing solids. The concentration of flowing solids would be expected to be different from that of upward flow in the same line and would probably vary across the height of the pipe. A brief study of an expansion and subsequent contraction in the line was also planned in an attempt to understand the sudden large increase in pressure drop observed in part I when the reverse flow rate fell to about 23 litre/min. and the retainer was full of solids.

It was decided to investigate the effect of pipe diameter by using two sizes and to use silver sand and ballotini as solids. These had fairly similar densities but were quite different in shape characteristics. Particles of about the same size were used for both materials so neither the effects of particle size nor of solids density were investigated.

5.3. Review of studies in solids conveying by pipeline.

The pipeline offers a simple and cheap method for cross-country transportation of minerals in bulk

between two fixed points and this has stimulated many studies in horizontal solids-liquid conveying.

Miss Blatch (34) made the earliest detailed investigation in 1906.

One of the earliest recorded commercial applications was to convey coal over 660 yards from the Thames to Hammersmith Power Station in 1913 (35). Between these early events and the present day, a considerable literature on horizontal pipeline conveying has appeared. This has ranged from purely commercial considerations (36) to empirical equations for predictions of transitions in flow behaviour (37).

Durand (38-41) has made a most comprehensive investigation into the flow properties of a wide range of particle sizes of solids with specific gravities about 2.6. Durand studied both horizontal and vertical flow and his investigations were later extended to cover particles of different densities by Worster (42). Abbott (43) gives a comprehensive list of references on horizontal flow.

Studies of vertical conveying - invariably vertically upward - are much rarer although vertical conveying was used to lift gold bearing gravel in America in the eighteen-fifties. In addition to the work

of Durand and Worster, Gliddon (44) studied exclusively vertical flow for a wide range of particle sizes and densities in 1 inch and 2 inch pipes. Maude and Whitmore (45) attempted a more theoretical description of flow behaviour and their predictions for 20 to 40 μ particles indicated increasing solids concentration on approaching the tube walls. Gliddon working with coarser particles found the opposite to be true while Sergé and Silberberg (46) found that solids were concentrated in an annulus some distance from the pipe walls. This variety of experience is illustrative of the complex nature of solids-liquid conveying and few correlations are applicable outside the sphere of their derivation.

However, Gliddon and Worster agree on a simple relationship for particles not less than 0.02 inches diameter in vertically upward conveying. In conveying these, it is found that a frictional pressure drop to velocity relationship is produced which is identical to that with water only flowing at the mean flow rate of the solids-liquid slurry. To obtain the frictional pressure drop, the pressure drop due to the static head of liquid and solids is subtracted from the absolute change in pressure.

With smaller particle sizes, this simple

relationship underestimates the pressure drop at very low velocities. The divergence increases with increasing solids concentration, decreasing flow rate, and decreasing particle size. The curves produced by Gliddon's studies are all similar in shape to the pressure drop against mean velocity curves obtained with water only in the line.

In all the preceding studies of both horizontal and vertical pipeline flow, the reported solids concentrations never exceed 40 percent by volume (excepting studies on very fine particles) since the experimenters have recognised that this limit avoids incursions into the different regime of dense phase flow.

Two studies of solids-liquid dense phase flow have been reported. Hancher and Jury (48) and Hershey and Peebles (49) both studied the vertical flow of settled ion-exchange resin in comparatively wide pipes at very low flow rates. They recognised that, with dense phase flow, the solids particles would be in continuous contact with surrounding particles and that pressure could be transmitted through the solids without affecting the liquid pressure. When solids moved in dense phase flow, any solids pressure would result in frictional losses between the solids and the pipe wall. The frictional losses would

depend on the solids pressure and on a coefficient of sliding friction.

Both Hancher and Jury and Hershey and Peebles attempted to measure the coefficient of sliding friction. By using wide tubes and vertically downward flow, they were able to measure directly the force exerted by solids friction on a plate suspended in the dense phase flow. However, solids occupied only a short length of pipe and this complicated the analysis since solids pressure varied across this short length. Use of ion-exchange resin was assumed to introduce a further complication.

The resin beads at the walls of the pipe could be distorted by solids pressure thereby allowing liquid pressure to be transmitted to the walls through the solids and augmenting the frictional losses.

Hancher and Jury derived only one very approximate measurement of a coefficient of friction. Hershey and Peebles found that the coefficient of friction varied with solids velocity over a range of very low velocities.

There have been a number of studies in solids-gas conveying in dense phase flow. The earliest appears to be that of Berg (47). Wen (31) is also reported as having investigated horizontal flow quite comprehensively. His observations of drag coefficient and of the lack of

effects of particle shape on friction losses bear similarities to the results of this study.

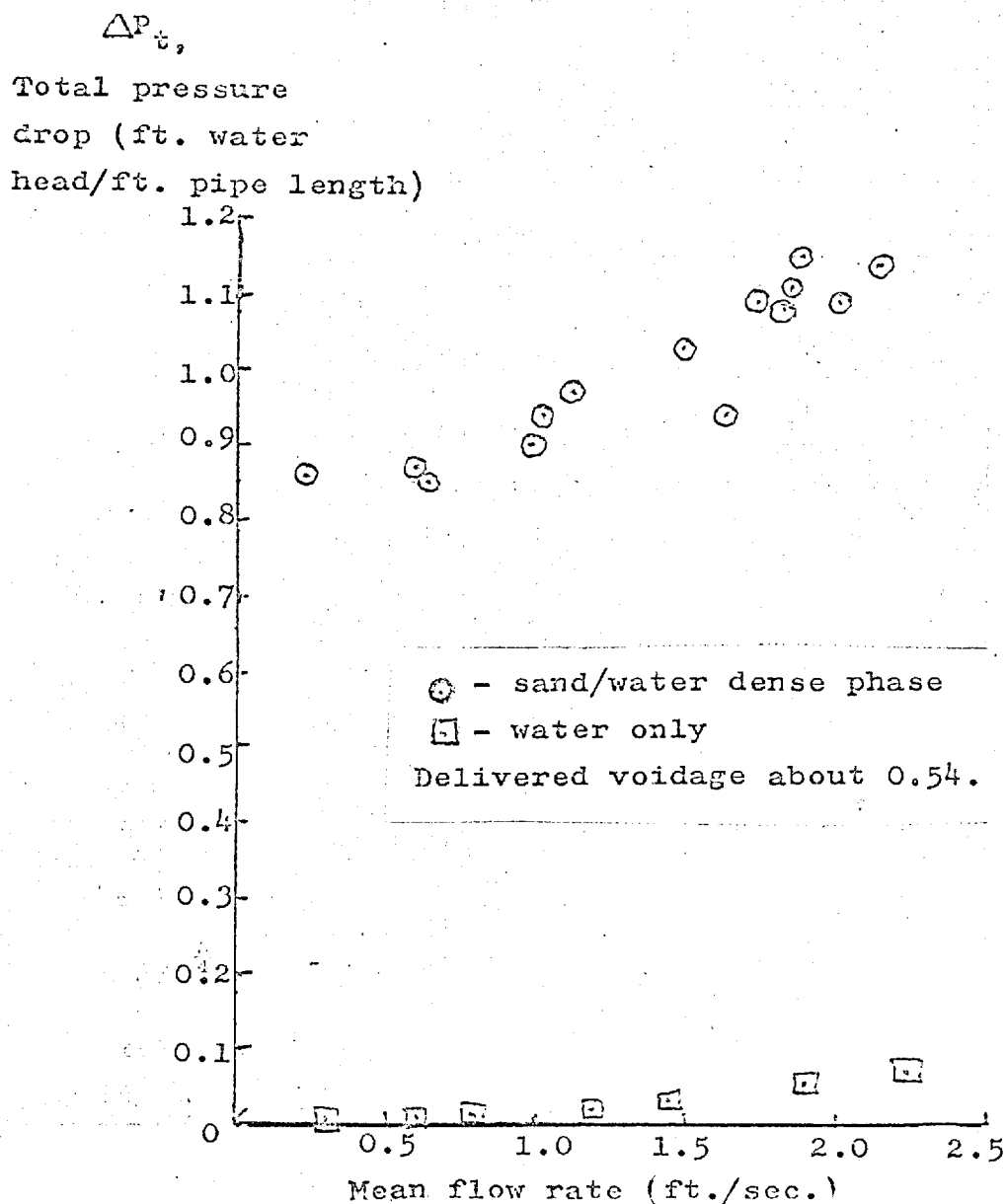
5.4. Observations of dense phase flow prior to this study.

In operating the pilot plant used in part I, a certain amount of qualitative information on dense phase flow was accumulated. A lower limit for dense phase flow rates through the retainer was observed and it was found to be especially difficult to carry dense phase flow through flexible P.V.C. lines. When this was attempted, a high pressure drop occurred and the solids moved forward in short spurts in a manner very similar to that described by Rose et al (4) with solids-gas dense phase flow. While there was some pulsation in flow through polyethylene and glass lines it was much less marked and only occurred temporarily after flow had been stopped.

One brief experiment had been carried out on the pressure drop in vertically rising dense phase flow through a $\frac{1}{2}$ inch polyethylene line at mean flow rates up to a little over 2 feet/sec. No measurement of the transmission solids concentration could be made but subtraction of a solids static head estimated from the delivered solids concentration gave results which could have been interpreted as reasonably coincident with the pressure drop caused by water only. (fig. 30).

Fig. 30.

Variation of total pressure drop with mean velocity of silver sand and water in vertically rising dense phase flow through a $\frac{1}{2}$ inch polyethylene line.



6. EXPERIMENTAL.

6.1. Planning of the Experiments.

6.1.1. Measurement of variables.

Measurement of delivered solids concentration could have been done either by weighing the solids output over a known time interval or by measuring the solids volume. Measurement of solids volume seemed preferable since the total volume of solids and water could also be measured and used to give the mean flow rate through the line. Solids volume could be reproducibly measured in the same way as in part I by briefly fluidizing the collected solids and measuring the free-settled volume.

The measurement of solids concentration in the vertically rising flow condition presented more problems. The measurement could be approached in three ways. Either the solids concentration could be measured directly or the absolute velocity of the liquid or the absolute velocity of the solids could be measured. (Knowing the volume of solids or liquid delivered in a given time, the concentration of that phase in the line can be inferred from its absolute velocity.) To measure either of the absolute velocities required tagging of some sort and introduction of the tagged material to the flow stream

without significantly disturbing the flow. Since the tagging would have had to be detected at two points in a steel pipe, a radioactive tracer seemed to be the only suitable type. Use of a liquid tracer would have required rigorous control over the liquid inventory of the system with the attendant complications in apparatus design. Use of a solids tracer would have required complex equipment to introduce the tracer slug to the test line. It was therefore decided to examine methods for direct measurement of solids concentration in the line.

The simplest way of measuring solids concentration in the flowing stream appeared to be to measure the attenuation of a beam of radiation passing through the flowing solids. Since the planned pipe diameters were less than an inch, a Bremsstrahlung source seemed to give the most suitable emission energy for diametrical attenuation (52). However, Bremsstrahlung radiation is a continuous wide band of energies and absorption of the low energy end would have occurred much more readily than of the high energy resulting in a complex relationship between transmitted radiation and the thickness of the absorbing media. The presence of pipe walls could completely alter this relationship.

The use of a γ -radiation source was next considered. This could either be attenuated axially in a suitably short length of vertically rising flow or be attenuated in a shallow angled traverse of the pipe. The variation of path length resulting from this shallow angle traverse would have required very fine collimation of the radiation beam and consequently a source of prohibitive size. The short vertical pipe section for axial absorption of γ -radiation was therefore adopted. This technique was used previously by Rhodes et al (51). The error in the measurement of densities is at a minimum when μm , the product of the mass absorption coefficient and the mass of the absorbing path (dimensionless), is about 2 ~~though~~ the minimum is shallow and an order of magnitude variation acceptable. (52). A value of $\mu m = 2.3$ was chosen corresponding to absorption of 90 percent of the unobstructed beam. This corresponds to an approximate path length of 22.1 cm.

The quantity of solid in the test section would be determined as a cumulative "depth" through which the collimating beam passed according to the equation:

$$\log_e (I_{sw}/I_{ov}) = -\mu_s \rho_s x_s - \mu_w \rho_w x_w$$

where I_{sw} = measured intensity of radiation with dense phase flow in the test section,

I_{ov} = measured intensity of radiation with a vacuum in the test section,

μ_s = mass absorption coefficient of the solids,
(cm^2/gm_s),

μ_w = mass absorption coefficient of water,
(cm^2/gm_w),

ρ_s = density of solids, (gm_s/cm^3),

ρ_w = density of water, (gm_w/cm^3),

x_s = depth of solids in the test section, (cm.),

x_w = depth of water in the test section, (cm.),

Since the depth of water plus the depth of solids was a constant in the test section, the equation would be linear.

Measurement of pressure drop across a straight vertically rising section of pipe-line was simple. The presence of a large concentration of solids would be expected to assist in smoothing the flow, but in any case 45 - 50 pipe diameters of undisturbed flow was allowed in the design before the first pressure tapping, as

recommended for complete smoothing of liquid flow (30).

6.1.2. Design requirements.

The material for the test line had to be sufficiently hard to withstand any abrasive action of solids particles and the corrosion of mains water. It had also to be of precision bore. Drawn tube of type ~~321~~ A.I.S.I. stainless steel made by Tube Investments was chosen. To give the minimum of changes in test line diameter stainless steel Ermeto pressure couplings (Ermeto Ltd., Maidenhead, England) were selected for use in the test line.

The test rig was planned to allow runs of at least 100 seconds duration in a $\frac{3}{4}$ inch line at velocities of up to 6 feet/sec. or 12 feet/sec. in a $\frac{1}{2}$ inch diameter line. This required a feed vessel with a capacity of 13 gallons. It was considered impractical to provide the storage capacity or the quantities of solids which would have been required for a 1 inch test line. A lower limit of $\frac{1}{2}$ inch pipe diameter was used giving a particle to pipe diameter ratio of about 1:34 for 40 mesh solids particles (30). Particles of about 40 mesh were chosen mainly because this was the size used in the earlier experiments and because it was sufficiently coarse to retain

particulate characteristics (41).

Only an order of magnitude estimate of the pressure drop likely to be encountered over the system could be made from the previous experience of dense phase flow in Part I. It was decided to use available pumps, at least in the first instance, but in view of the possibility that pressure drops might be higher than estimated, the pressurised feed vessel, the associated water input line, and the valves opening onto the feed vessel were specified and tested to 200 lb./in.²

6.2. Experimental plant.

6.2.1. The main plant.

The experimental plant is shown in fig. 31 and 32. The central feature was the pressurised feed vessel, F, which was an 8 inch diameter by 7 foot mild steel pipe of $\frac{1}{4}$ inch wall thickness shut off at both ends with removable blank flanges. This vessel had a nominal capacity of 15 gallons. A concrete conical funnel was built up on the bottom blank flange to allow complete drainage of solids without dismantling the vessel.

The $\frac{1}{2}$ inch and $\frac{3}{4}$ inch test lines were connected in turn at the base of the pressurised feed vessel. Flow to the test lines first passed through an 'M' type Saunders valve, 8. This is a 90° action ball valve causing negligible deviation or obstruction of the flow when open. The valves in position 8 for both lines were fitted with stainless steel balls and suffered negligible wear from silver sand but were rapidly scored by ballotini. Flow in the test line was initially carried in copper tubing of the nominal size of the line and a 'T' junction was inserted in this immediately below the feed vessel for drainage and to admit mains water through valve 9 to flush the test line of solids. This copper tubing carried

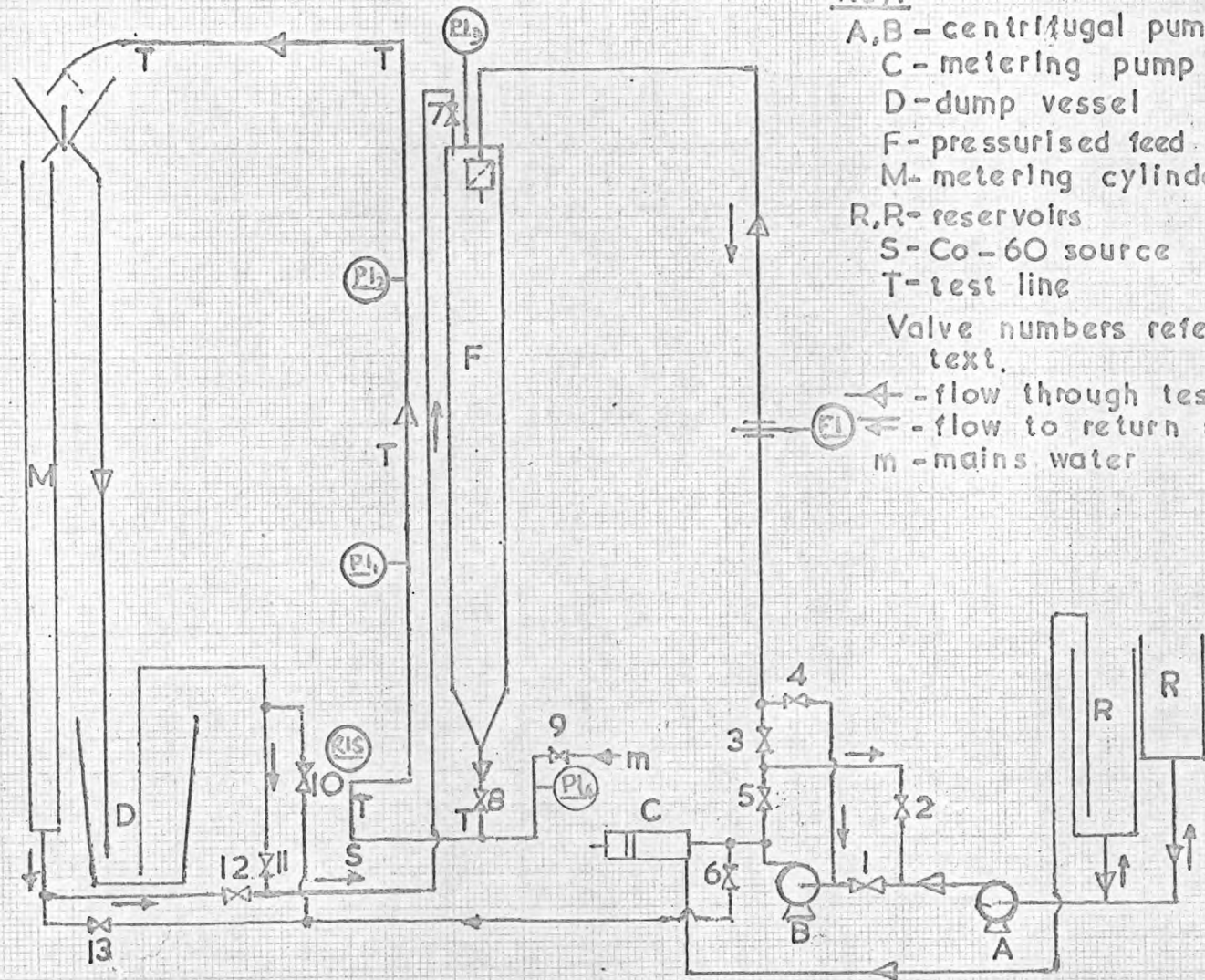
FIG. 31.
DENSE PHASE FLOW APPARATUS.
FLOW SHEET.

Key.

- A,B - centrifugal pumps
- C - metering pump
- D - dump vessel
- F - pressurised feed vessel
- M - metering cylinder
- R,R - reservoirs
- S - Co-60 source
- T - test line

Valve numbers refer to text.

- flow through test line
- flow to return solids
- m - mains water



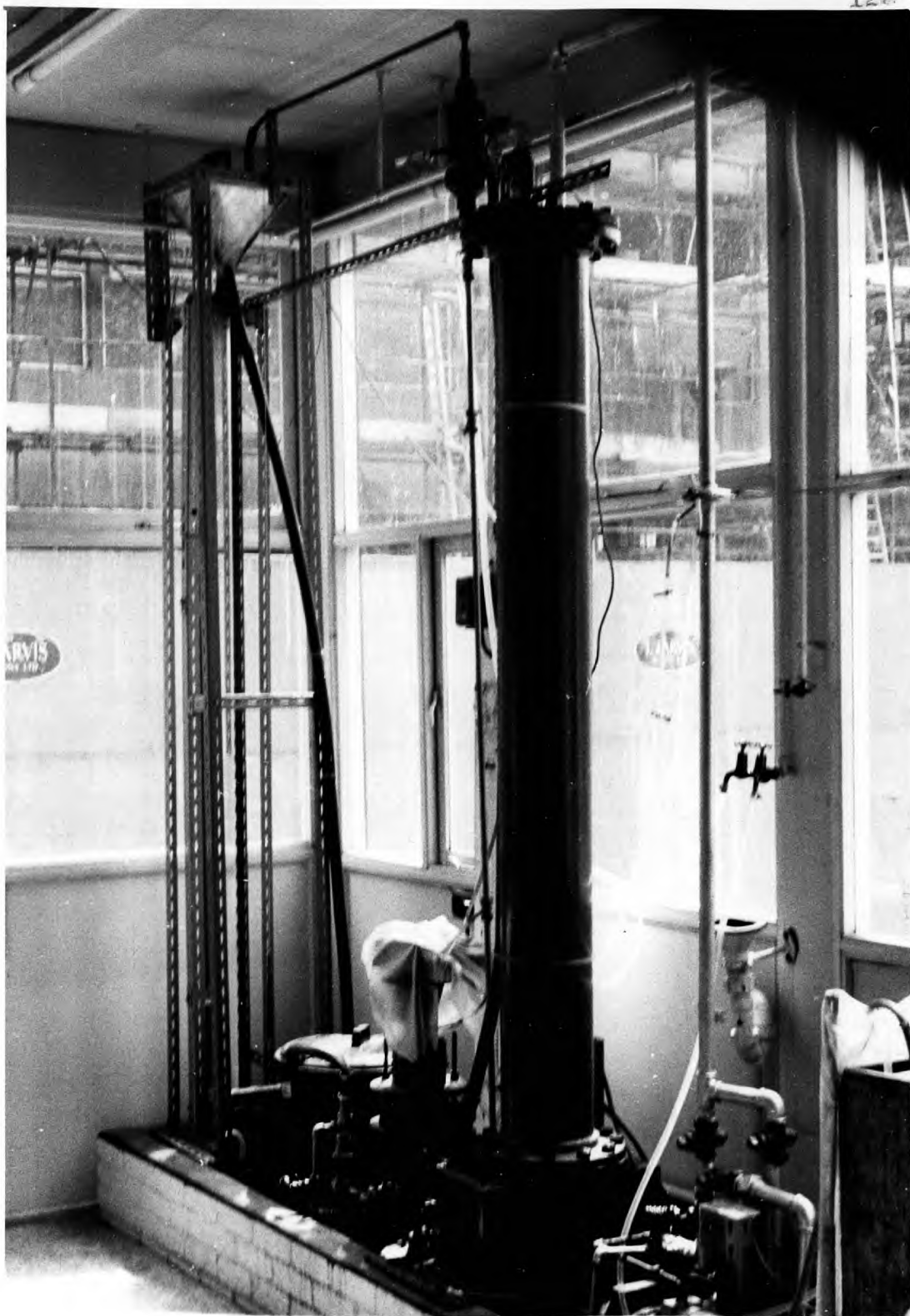


Fig. 32.

Dense phase flow apparatus.

flow to the entry point to the density gauge. Here the line changed to precision bore stainless steel tubing. The change occurred at a right angle stainless steel Hermeto coupling. After the density gauge (see section 6.2.2.1.) and ~~and~~ short horizontal section, flow entered the main rising section of about 9 foot length. Approximately the central 4 feet of this were used to measure the pressure drop of vertically rising flow. At the top of this vertical section, a further right angle coupling returned flow to copper tubing and after a short horizontal section and a shallow curve, flow was passed to either side of a dividing cone by a swinging flexible pipe. (For the first 53 runs out of a total of 70 with silver sand in the $\frac{1}{2}$ inch line, there was a shallow bend of about 5 inches radius at the top of the vertically rising section.) The swinging pipe was attached loosely over the outlet of the copper tube and consisted of about a foot of 1 inch bore neoprene reinforced with imbedded steel rings.

From the dividing cone, solids passed either to the metering cylinder, M, or to the dump vessel, D. The metering cylinder was an 8 foot long by 4 inch diameter glass cylinder of uniform bore. The dump vessel was a plastic dustbin of about $15\frac{1}{2}$ gal. capacity.

Only one major modification was made to this plant. For the last seven runs with ballotini in the $\frac{1}{2}$ inch line, the top 20 inches of the vertically rising section were replaced by an 8 inch length of 3 inch diameter mild steel pipe connected to the $\frac{1}{2}$ inch line by standard reducers and bushes. This is shown in fig. 33.

Three pumps produced flow in the test line by pumping water into the top of the pressurised feed vessel. Pump **B** was a centrifugal capable of developing 24 lb./in.² The four valves, 1 - 4, round this pump could direct flow in either direction between the feed vessel and the main tanks. Pump **A** was an identical pump used in series with pump **B** to boost the pressure in the feed vessel to 48 lb./in.² The maximum pressure could be developed at the highest flow rates used in either of the test lines. Flow rates were controlled by the 1 inch valve, 5, immediately downstream from the output of pump **B**. Pump C was a metering pump (maximum capacity about 7.8 gal./hr.) to give flow rates in the test line below those which could be accurately or easily produced by the centrifugal pumps. The main water feed tanks were linked together and had a nominal total capacity of 17½ gallons.

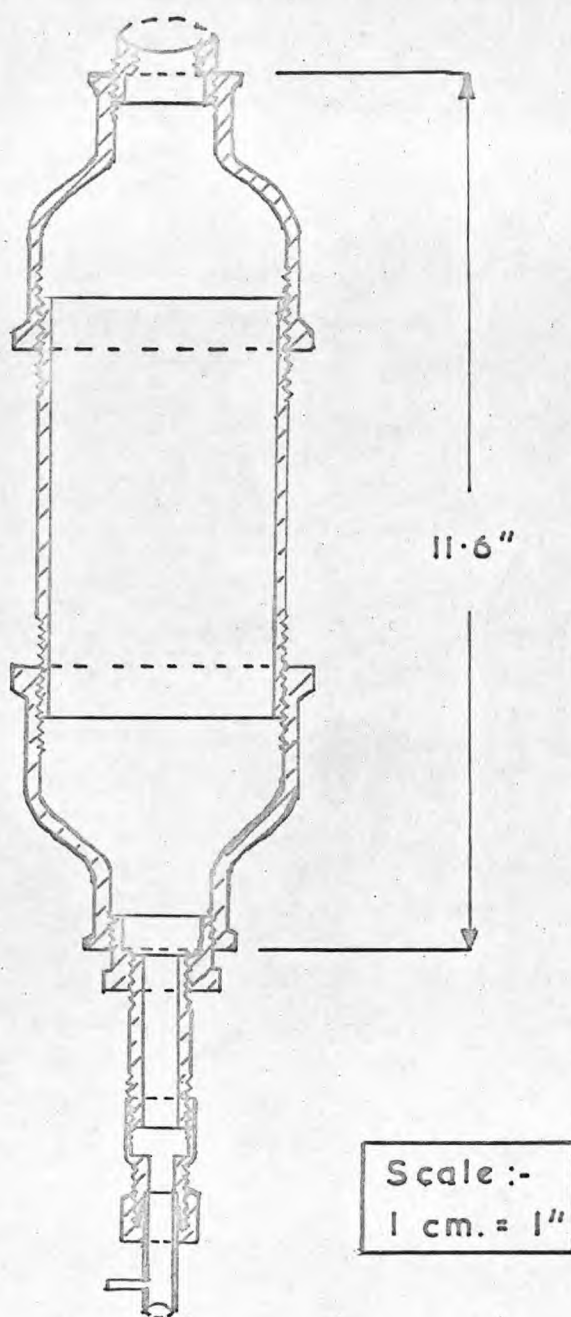


FIG.33 - SECTION THROUGH VERTICAL
AXIS OF EXPANSION-CONTRACTION IN 1/2"
LINE.

Solids were returned to the top of the feed vessel from the metering cylinder and the dump vessel through 1 inch bore P.V.C. lines. The suction side of pump A was used to move the solids. Since the maximum pressure drop which could be produced in this way was only 8 lb./in.², a small amount of water from the output side of the pump was added to the dense phase flow close to the dump vessel and the metering cylinder. This resulted in dilute phase transport of the solids and avoided the high pressure drops which had been experienced with dense phase flow in P.V.C. lines.

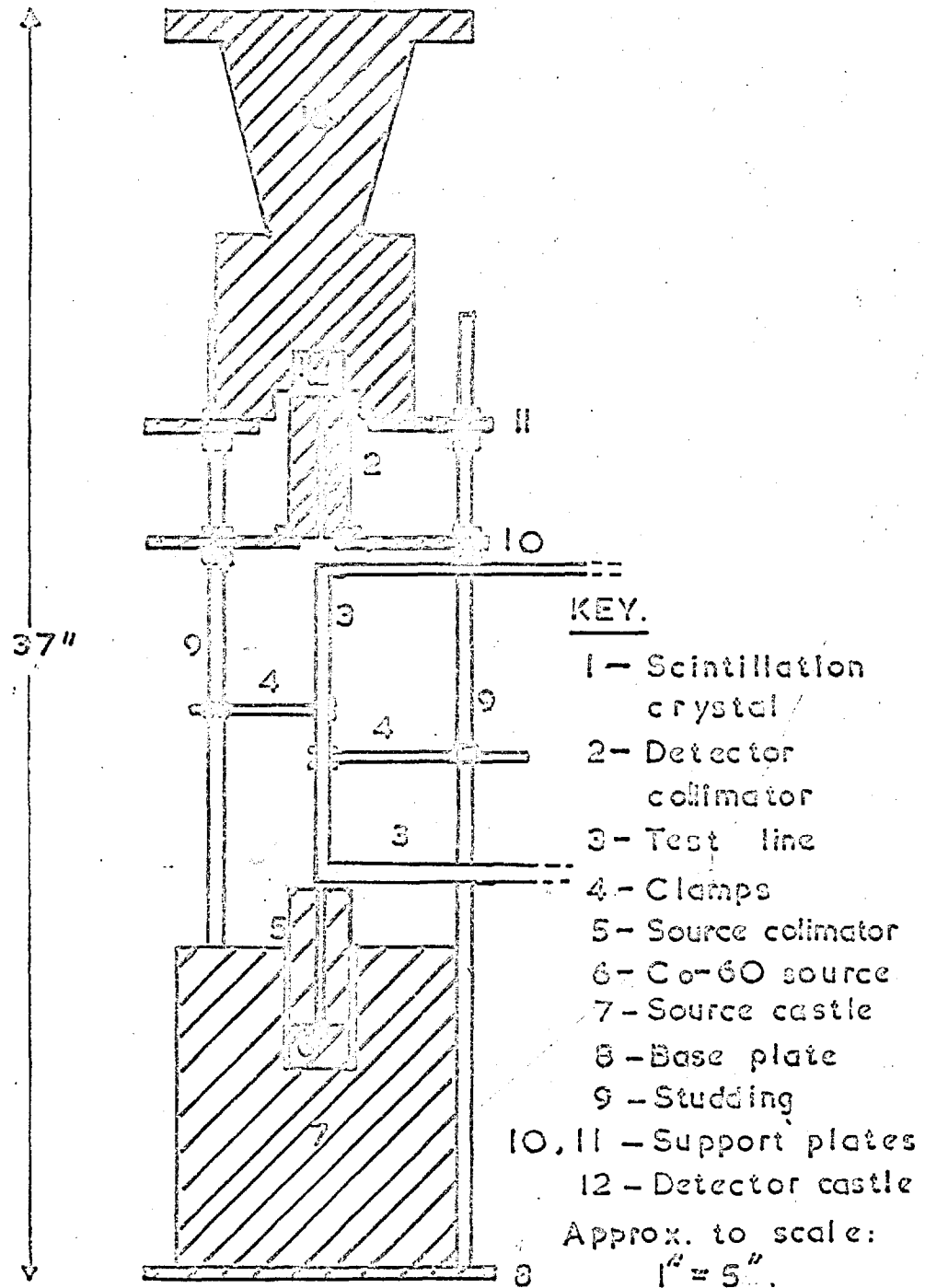
Water could be easily disposed off from the system by allowing either the main tanks or the dump vessel to overflow into the drain trough in which the apparatus was built.

6.2.2. Instruments.

6.2.2.1. Density gauge.

The layout of the density gauge is shown in fig. 34. The 10 mC Co-60 source was sealed inside a cylinder of 0.69 cm. diameter and 0.81 cm. long, 6. It was mounted with the axis vertical in the central hole of a brass cylinder which fitted into the 5.07 cm. diameter hole in a cylindrical lead castle. A collimating

DENSITY GAUGE
AXIAL VERTICAL SECTION.



cylinder of 9 cm. length was inserted on top of the brass cylinder locating the source. This collimator was a lead filled brass tube with brass end plates in which were fitted the ends of the 0.5 cm. I.D. copper tube collimator.

The castle, 7, was mounted on a circular steel base plate, 8. Three pieces of $\frac{1}{2}$ inch studding were screwed into this base plate so as to touch the outside of the castle and were drilled, tapped, and bolted into the cast iron outer shell of the castle. Two further steel plates, 10, 11, with drilled $9/16$ inch holes concurrent with the tapped holes of the base plate were mounted above the castle and located by nuts on the studding. The lower of the two plates, 10, supported the detector collimator, 2. This plate had a central $\frac{3}{4}$ inch hole and surrounding this were three $\frac{3}{8}$ inch holes in which the detector collimator could be rigidly located by using washers and 2 BA bolts to give some latitude in the position.

The detector collimator was 9 cm. long by 1.8 cm. diameter and the collimating slit was 0.5 cm. in diameter. It was of similar construction to the source collimator but had in addition a brass collar through which the locating bolts passed. In setting up this collimator, its position on the steel plate and the tilt of the steel

plate were adjusted until the radiation flux leaving this collimator was at a maximum. These collimating arrangements ensured a maximum difference in path lengths passing through the test section and air only of 0.025%. Lead bricks were placed around the detector collimator to reduce the background and interference from scattered radiation.

The detector was also mounted in a lead castle, 12, and this was supported on the top steel plate, 11, and held in place by its weight. The steel plate, 11, had a $2\frac{3}{4}$ inch central hole and the top of the detector collimator passed through this to terminate close to the detector crystal, 1. The IDI 661 detector comprised an 11/16 inch diameter by 1 inch thick type 2004 thallium activated sodium iodide crystal and an EMI 9254B photomultiplier. The cathode follower was a Dynatron Channel Probe Unit NI 14 set at maximum sensitivity with a paralysis time setting of 200 μ sec. (This is a pulse generator unit sending out a standard pulse for every pulse received above a particular voltage determined by the sensitivity setting.) The output from this was passed to an Ecko Automatic Scaler Type N530F.

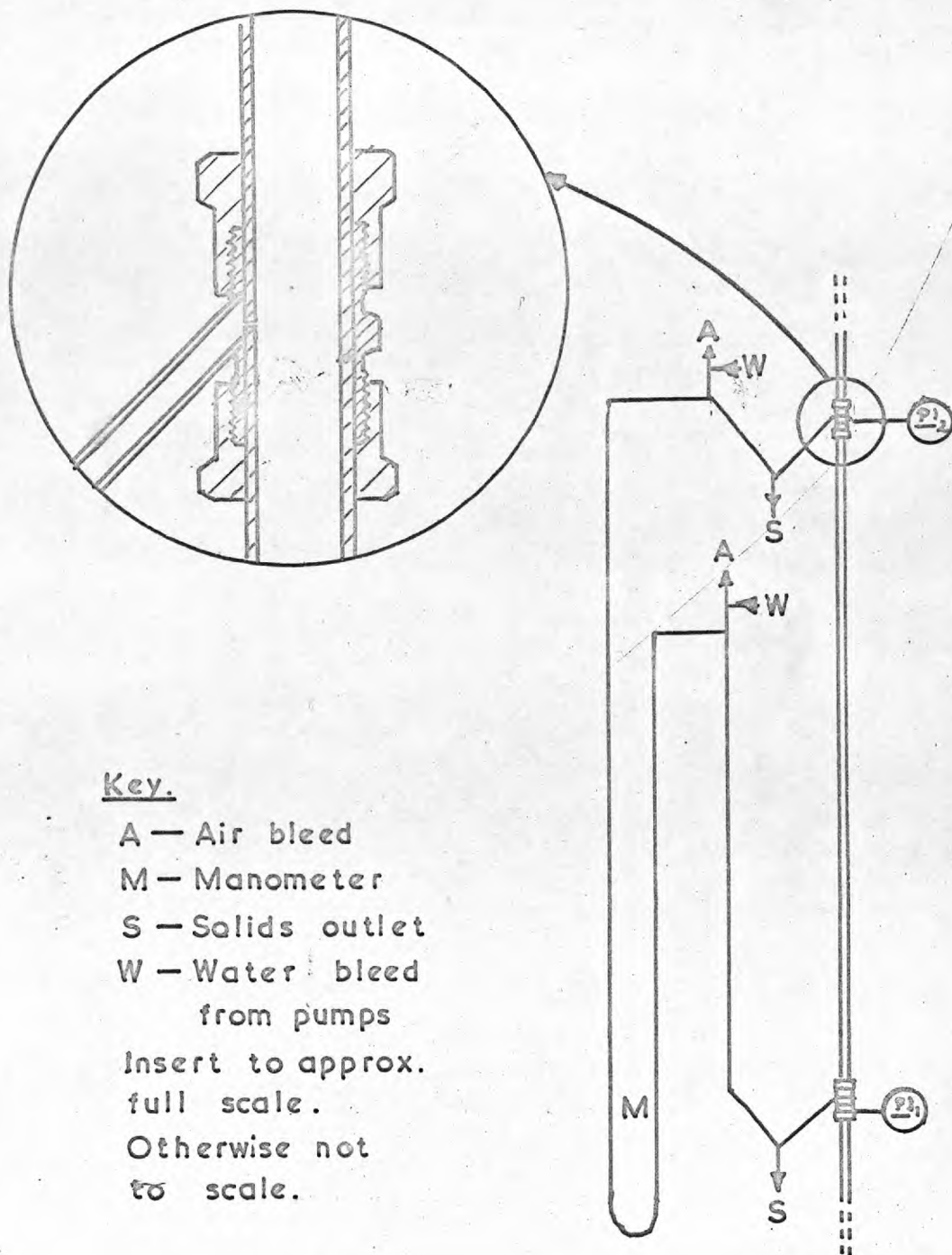
The approximately 22.1 cm. long test section of pipe was located while empty by moving it slightly till the radiation reaching the detector was at a maximum. It was rigidly located by four specially shaped clamps, 4, attached to the studding of the support structure. The Ermeto elbows would have given a curved boundary to the ends of the column of flowing material in the test section. To avoid this they were drilled flat with a star drill before assembly.

6.2.2.2. Pressure measurement.

The pressure tapping holes, PI_1 and PI_2 , were 1/32 inch in diameter in order to be not larger than one sixteenth of the tube diameter. (This is the upper limit recommended by Allen and Hooper (54).) They were drilled with a stainless steel bit with a tight fitting brass piston inside the tube to prevent burrs on the tube wall.

Brass Ermeto couplings were drilled out to give a loose fit over the tube. A hole was drilled into the coupling wall to take a 1/4 inch O.D. copper tube which was soldered in place. (Fig. 35). One of these couplings was located and tightened over each of the 1/32 inch holes in the tube wall. By using brass sealing rings on these couplings, it was possible to remove them subsequently since the brass did not cut into the stainless

FIG.35. - PRESSURE MEASUREMENT ACROSS
VERTICALLY RISING FLOW SECTION.
INSERT OF AXIAL SECTION OF PRESSURE
TAPPING.



Key.

- A — Air bleed
 - M — Manometer
 - S — Solids outlet
 - W — Water bleed from pumps
- Insert to approx.
full scale.
Otherwise not
to scale.

steel. The two tapplings were connected across an 80 cm. mercury-water manometer. (Fig. 35). Both the $\frac{1}{4}$ inch P.V.C. leads to the manometer had points where solids could be removed and other points to introduce water bleeds from the main pumping system.

A gauge to measure the actual pressure on the lower of these two tapplings, PI_1 was attached after the first 30 runs with sand in the $\frac{1}{2}$ " line. The gauge pressure measured here was designated P3.

Pressure measurement was made throughout of the pressure in the feed vessel (designated ODP) and, later on in the experiments, another pressure gauge was introduced at the 'T' junction in the test line below the feed vessel (designated P4). All pressure gauges were of the Bourdon type.

6.2.2.3. Flow rates.

The flow rates through the test line were determined by measuring the volume of sand and of sand plus water delivered to the metering cylinder in a known time. Flow was first started to the dump vessel. When adjusted to the required overall pressure drop, flow was diverted to the metering cylinder by the swinging pipe. The diameter of the cylinder was previously measured by withdrawing measured volumes of water of about one litre

each and plotting the level against the total amount withdrawn. The diameter was found to be constant and 1 cm. of column length to be equivalent to 0.0188 gallons.

The timing of a few of the early runs was done with a stop watch operated by the other hand from that operating the swinging pipe. This was sufficiently accurate with an error of about 0.2 seconds at most if the duration of flow to the metering cylinder was more than a minute. (Equivalent to velocities not exceeding about 5 feet/sec. in the $\frac{1}{2}$ inch line.) It was superseded before any runs were carried out with both centrifugals in series which gave velocities in excess of 5 feet/sec. The subsequent equipment incorporated a 'reed' switch (magnetic proximity switch) mounted on a boom close to the swinging pipe. A magnet was attached to the swinging pipe and closed the reed switch when the swinging pipe was in the position to direct flow to the metering cylinder. This completed the circuit to activate a Venner Mark II Timer (Venner Ltd., New Malden, Surrey).

This instrument was designed to time fractions of a second. When the timing circuit was closed, a low inertia hand with a one second sweep picked up the continuously running mechanism. The dial on this hand was graduated in hundredths of a second and could be accurately read to

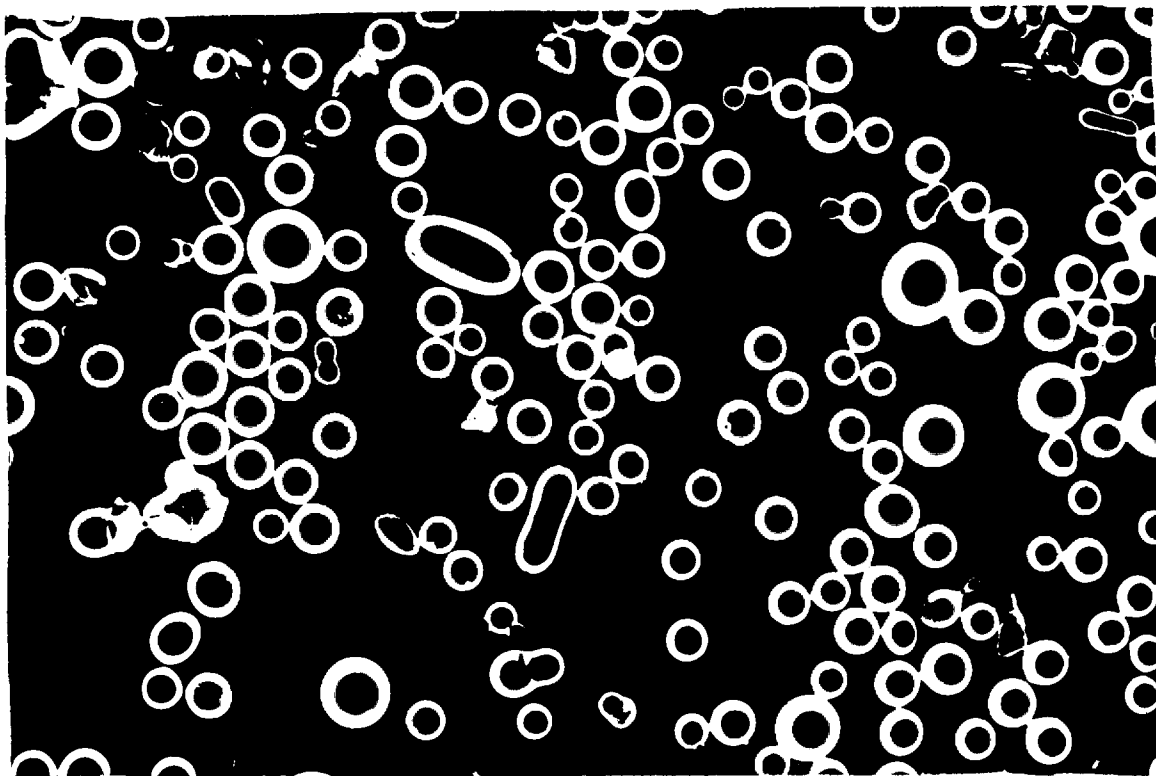
1/1000 second. To record seconds, the timer closed contacts in a separate circuit once every sweep of the second hand and these pulses were counted by a relay pulse counter.

6.2.3. Characteristics of solids materials.

The silver sand was the same material as used in the Part I experiments and described in section 2.1.2.

The glass ballotini were silicone coated soda glass of nominal 0.3 to 0.4 mm. (about ~~36~~ + 52 B.S.S. mesh.)

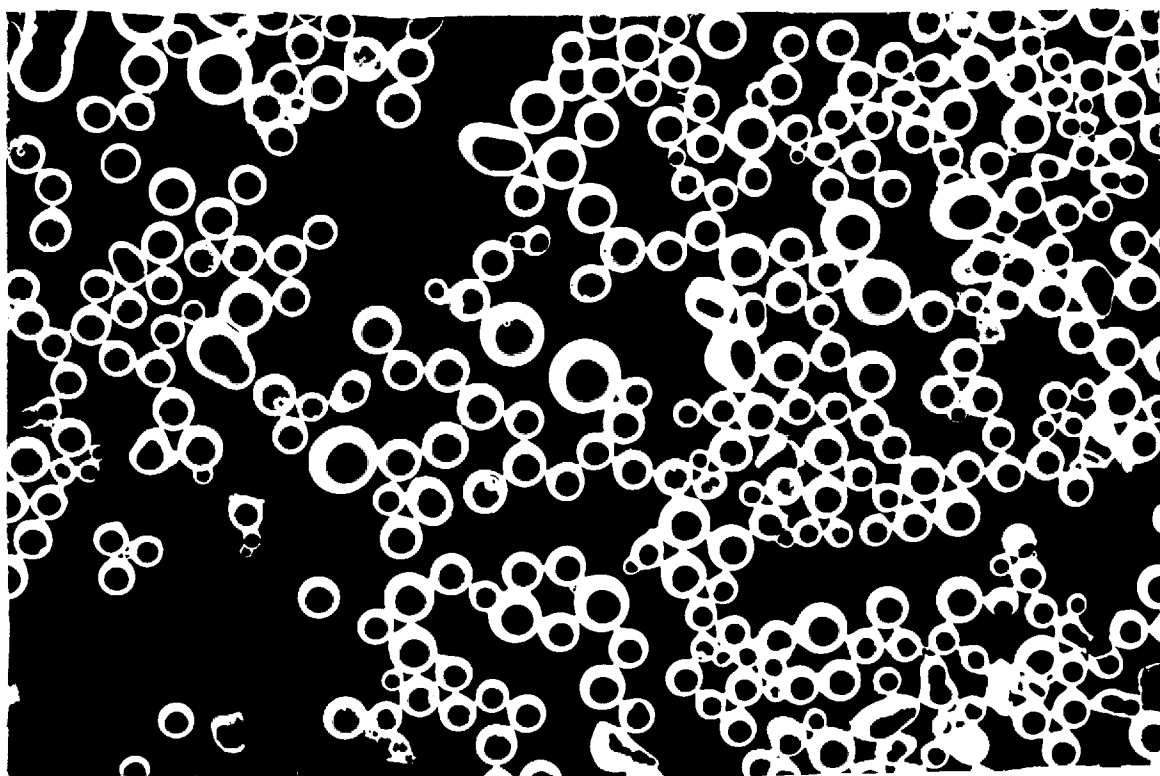
Fig. 36 shows a sample of the ballotini with a magnification of $\times 18$. 100 particle diameters were measured to estimate the size distribution in the same way as for ion-exchange resin described in section 2.1.2. The absolute density of the ballotini was found to be 2.4978 gm./ml. The free-settled voidage was determined as being 0.4395 and the tap-settled voidage as being 0.385. Full details of the characterisation of fluidized ballotini and size distribution are given in Appendix 1.



Unused.

Fig. 36. Ballotini. Negative photographs, magnification $\times 18$.

After use throughout the dense phase flow experiments.



6.3. Experimental method.

6.3.1. Flow Control.

In practice, flow through the test lines was usually started with only water present in the lines since readings of the density gauge with water in the test section always preceded a dense phase flow run. However, flow could be re-started after dense phase flow had been allowed to settle for an indefinite period in the test line. On re-starting flow with the centrifugal pumps, the overall pressure drop rose to the maximum available from the pumps. The solids tended to move in slight spurts for a variable time of anything from a few seconds to several minutes and then re-established the original flow conditions. With the positive displacement metering pump, the overall pressure drop rose to as much as 100 lb./in.² with much fluctuation and sudden spurts of flow. Again flow ultimately re-established the original conditions though with the positive displacement pump up to twenty minutes could first elapse. With both types of pump, there was a tendency for the interval before original flow was re-established to vary directly with the period of settling in the test line.

This tendency for a longer delay in re-establishing

the original flow conditions after a long period of settling should not be confused with the complete absence of flow and associated high overall pressure drop which arose if air was present in the test line in the interstices between the settled solids. In this situation, the solids did not begin to move until the flow of water through the interstices had expelled most, if not all, of the air. Again, flow eventually returned to the original conditions.

Wide variations were observed in the pressure drops when flow was re-started using the metering pump after a period of settling of solids in the line. Even two consecutive runs with the same flow rate and after equal periods of settling time rarely gave similar pressure drop patterns. This suggested that there would be no advantage in providing larger positive displacement pumps to study the transitory flow behaviour of solids after settling at higher flow rates.

Under stable conditions, the centrifugal pumps were quite satisfactory and provided a substantially constant flow rate without requiring any adjustment to the flow regulating throttling valve.

6.3.2. Manometer technique.

The measurement of pressure drop by manometer gave accurate readings at negligible cost. However, movement of mercury levels in the manometer required removal of up to 3 ml. of water from the dense phase flow slurry. If the flow rate was gradually increased in the test line, the pressures registered by the manometer never exceeded the static head of solids and at high flow rates became negative. These were clearly erroneous and may have arisen from blockage of the lower orifice to the manometer through which water had initially to pass from the dense phase flow. This plug of solids would then presumably almost cease to transmit pressure at the lower tapping point. At the upper tapping point and in the absence of a similar plug, pressure would be transmitted normally to give the observed negative readings on the manometer. By using the water bleeds to the manometer (fig. 35) to pre-set the manometer rather higher than the expected pressure drop, consistent and increasing (with increasing flow rate) readings were obtained. This pre-setting could be overdone and if an excessively high pressure drop was initially set on the manometer, a high and irreproducible pressure reading was obtained.

Occasionally, the pressure tapping holes became more permanently jammed with solids and the pressure across the manometer altered only slowly. This was unacceptable with high velocity runs and had to be cleared by alternate application of hydraulic pressure and suction to the tapping points.

6.3.3. Density gauge calibration.

After the density gauge had been installed and aligned but before the $\frac{1}{2}$ inch test line was located in the beam of the density gauge, a 22.1 cm. length of the same $\frac{1}{2}$ inch stainless steel tubing closed at one end by a piece of perspex sheet was used to derive the mass absorption coefficients of silver sand and ballotini.

The distribution of solids in the calibration tube was non-homogeneous. However, this was unimportant since the radiation beam was well-collimated. Also, only a very short section at the top of the calibration tube did not contain solids when the attenuation equalled that produced by dense phase flow in the test line.

A $\frac{1}{2}$ inch steel plate was inserted in the beam first above and then below the calibration tube and no difference was observed in the relative attenuation produced by the empty tube and the tube filled with water. This confirmed the theoretical expectation that

the attenuation caused by the steel walls of the couplings at either end of the test section in flowing conditions could be ignored.

To obtain the mass absorption coefficients, the intensity of measured radiation was noted with the tube empty and a small correction (multiplied by 1.002) applied for the attenuation caused by air. The calibration tube was then filled with water and the count rate noted. The path length and the density of the water were known and the mass absorption coefficient could be calculated. The solids mass absorption coefficients were found by introducing known weights of solids into the calibration tube which was otherwise filled with water. The calibration tube was located with the same clamps used with the test line. The weights of solids added to the calibration tube were varied widely to establish that the plot of $\log_e(I_{sw}/I_{ov})$ against x_s , the depth of solids, was a straight line. (See section 6.1.1.)

Attempts to add solids directly to the calibration tube frequently entrained air bubbles. This was particularly liable to happen when the tube was almost full of solids and produced a slight deviation at the high values of x_s in the otherwise straight line plot against $\log_e(I_{sw}/I_{ov})$. To avoid air entrainment, the calibration tube was connected by a short piece of rubber tubing to a

filter funnel and water added until the filter funnel was about half full. A known weight of solids was now sprayed onto the surface of the water in the filter funnel through the narrow orifice of a separating funnel. As soon as a layer of solids built up on the water surface, solids sank as individual air-free particles into the calibration tube. The final layer was easily sunk by the addition of a few drops of liquid detergent and stirring. This removed the anomalous deviation from a linear plot. The calibration details are given in Appendix 3.

Before the test section was used for the first time and subsequently after every removal of the test section from its supporting clamps, the length of the column of flow in the test section - the path length - had to be measured. This was done by alternately filling the test section with water and then removing the water with a water ejector. The total counts over two 100 second periods were noted for each state and the procedure repeated until three pairs of readings both with and without water were obtained. These were rejected and the procedure continued if any significant instrument drift seemed to have occurred during the collation. The path length could then readily be calculated

using the previously determined mass absorption coefficient for water.

6.3.4. Procedure for carrying out a run.

A sufficient quantity of solids was passed through the test line to give a readable level of solids in the metering cylinder. These solids were fluidized gently and the free-settled level noted together with the water level. The fractional second timer was zeroed and the reading on the seconds counting relay was noted. Flow from the test line was now directed to the dump tank and the solids flushed from the line by mains water applied through valve 9 to the junction in the test line below the feed vessel. Two 100 second counts were made of the detected radiation with water only in the test line (since the readings of the electronic counting equipment drifted slowly over a range of about 4%, varying principally with room temperature). The pump(s) for the run was then started with the pressurised feed vessel outlets both closed. Flow was started to the test line by opening valve 8 (fig. 31) at the bottom of the feed vessel and the desired flow rate adjusted with the throttling valve 5 by reference to the overall pressure drop. (The overall pressure drop indicated the flow rate as accurately as was required for these experiments and the flow rate measuring orifice plate between the

pumps and the feed vessel was not used.)

Now that flow was established at the desired rate in the test line, the manometer recording pressure drop across the straight vertically rising section of the test line was pre-set as explained in section 6.3.2. Next, the radiation detection equipment was started on a 100 second count and the flow was diverted to the metering cylinder automatically starting the timer. Readings were now noted of all the pressure measurements.

While the run was proceeding, the maximum number of 100 second counts of the radiation were made (except for very low flow rate runs where about six counts were made at intervals throughout the run). With the highest rate runs in the $\frac{3}{4}$ inch test line, the capacity of the feed vessel was only sufficient for some 70 seconds of flow and the time to measure 10,000 counts was noted - about 50 seconds - since the counter could not be set for less than 100 seconds. The pressure readings were noted periodically during the run since small fluctuations occurred and the average of each reading was assigned to that run.

Flow was returned to the dump vessel and the timer stopped when the metering cylinder was about four-fifths full (except at very low flow rates when

the volume collected had to be reduced to as little as 0.2 gal over $1\frac{1}{2}$ hours). Flow to the test line was stopped by closing valve 8, the test line was again flushed with mains water, and two further radiation measurements of 100 seconds were made with water in the test line. The water level in the metering cylinder and the readings of the timer and the seconds counter were noted. The solids in the metering cylinder were fluidized and the level of the free-settled solids noted. The solids were then returned to the feed vessel.

The flow rates of runs were randomised as much as possible and the experiments on a particular pipe size and solids material were continued until a reasonably continuous range of flow rates and pressure drops had been obtained. A number of runs were carried out in the vicinity of any suspected discontinuities in any of the pressure drops.

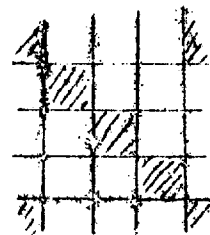
6.4. Calculation of results.

The results were calculated as the experimental runs were completed. To check these results and to extend the calculations, all the experimental readings were put on punched cards and a computer programme run on the Imperial College IBM 7090. A sample calculation is set out in Appendix 4.

Three forms of velocity appear in the results. Mean velocity is the mean linear velocity of solids and liquid calculated from the total volume delivered in unit time divided by the cross-sectional area of the pipe. Solids velocity is the mean linear velocity calculated from the absolute volume of solids delivered in unit time divided by the cross-sectional area of the pipe occupied by solids. Slip velocity has already been defined in section 3.5. This is mainly quoted as a percentage of solids velocity in the form of slip ratio.

It may not be immediately obvious that x_s , the linear depth of solids, divided by x_{pl} , the linear path length, is a direct measurement of the volumetric solids fraction, $(1-e_t)$. However, consider the case of a square

of side, say, three units. If every third unit is filled with solids, it can easily be seen that from all directions x_s/x_{pl} is $1/3$ and that the overall solids fraction is $1/3$. The same result is found if the model is extended into three dimensions.



In order to clarify the forms of pressure drop used to express the pressure drop over the vertically rising section, consider a section of pipe of height, h , filled with liquid of density, ρ_1 . When liquid is flowing upward in this section, there are two components of pressure drop. There is the frictional pressure drop due to fluid flow and a pressure drop due to the static head of liquid. The pressure drop due to the static head of liquid is $\rho_1 h$ (expressed in the conventional units of water head). The static head is the force which must be applied in moving the liquid through the pipe section against the force of gravity.

If solids particles of the same density as the liquid were now to be introduced to the pipe section, the liquid buoyancy would support the particles and the static head would be unaltered. If the density of these particles were now to be increased, a new element of

static head would arise equal to the difference in density between the solids and liquid, $(\rho_s - \rho_l)$, multiplied by the fraction of the volume of the pipe section filled with solids multiplied by the height of the pipe section. That is, the solids static head is $(\rho_s - \rho_l)(1 - e_t)h$.

Providing that the solids remain in suspension in the liquid, the solids static head is transmitted to the liquid by a drag force produced by the slip velocity of liquid past the solids. Hence, the solids static head appears in the observed water pressure at the base of the pipe section. This is the basic principle of fluidization.

Now, with the solids fractions of dense phase flow being of the same magnitude as packed beds, it might be assumed that at least some of the solids static head would be transmitted to the walls of the pipe. This occurs with stationary packed beds of solids and give rise to the phenomena associated with the angle of internal friction. However, in dense phase flow, the solids are moving upwards and no frictional force at the walls could act upward against gravity on the solids. Indeed, if such a force did exist, it would have to do

work in moving the solids against gravity.

In measuring pressure drop with vertically rising dense phase flow, the liquid static head is of no interest since its nature is well established. If the liquid frictional pressure drop, which appeared to be negligible (section 7.1), is ignored, the absolute pressure drop less the static head of liquid represents the total force exerted on the solids by the liquid. This is referred to as the "total pressure drop", ΔP_t . Since the manometer leads were filled with water, the pressure indicated on the manometer between the two tapping points on the section of vertically rising dense phase flow (fig. 35) does not include the static head of water in the test section. Effectively, it measures the total pressure drop as a head of mercury in water.

If the solids static head is subtracted from the total pressure drop, one is left with the force dissipated by solids friction. This is referred to as the "dynamic pressure drop", ΔP_d .

The pressure drop over the entire test line, ODP, is expressed as a "total" pressure drop. That is, only the static head of water is subtracted from the observed gauge pressure. The choice of this form of

pressure drop is arbitrary since the principal interest in this variable is in the transitions which occur. These are equally apparent if "dynamic" pressure drop is used with the static head of solids and the static head of liquid subtracted from the gauge pressure.

6.5. Experimental errors.

In counting radioactive disintegrations, the relative standard deviation from an observed count rate is given by $(C)^{-\frac{1}{2}}$ (53). Where several counting operations are involved and the result produced by multiplications or divisions of these counts, the relative standard deviation is given by:

$$\text{Relative standard deviation} = \left[\sum (C^{-1}) \right]^{\frac{1}{2}}$$

where C = total count of the individual contributions.

Using a single 100 second count of the radiation attenuated by dense phase flow and four 100 second counts of the radiation attenuated by water (regarded as one 400 second count) gives a standard deviation of 0.71% for solids fraction. This is the pattern of counting in the majority of the experiments though in many of the lower flow rate runs more than one 100 second count of the radiation attenuated by flowing solids were made giving improved accuracy. With runs at the flow rates of greater than about 6 feet/sec. in the $\frac{3}{4}$ inch line the accuracy would be appreciably reduced to a standard deviation of about 1.35% since the radiation attenuated by dense phase flow could only be counted for about 50 seconds. (The error caused by reading of the time to count 10,000 pulses to only 0.1 seconds is relatively unimportant.)

Measurement of the delivered solids fraction depended on the accuracy of measurement of solids volume in the metering cylinder. The reproducibility of a given solids level at the bottom of the metering cylinder was excellent but measurement of errors further up the column were reproducible only within a range $\pm 0.5\%$. In calibrating the metering cylinder for the two solids, the levels were repeatedly measured and fluidized but the weighing associated with the calibration was only accurate to about $\pm 0.5\%$. That is, in comparisons between the delivered voidages of the two materials an error of $\pm 1\%$ could exist.

Using the Venner Timer, timing of flow to the metering cylinder was read to $1/100$ sec. and the shortest period timed was rather over $3\frac{1}{2}$ sec. The timing in this case would have been in error by less than $\pm 0.15\%$. For the majority of runs, timing error depended on the accuracy of mains 50 cycles. The Venner Timer was checked against a caesium clock one second time signal and was at worst accurate to 0.1 sec. in 1 minute or $\pm 0.17\%$. Water levels in the metering cylinder could be read accurately to ± 0.5 mm. and the change in level was never less than 50 cm. (except for a few runs at very

low flow rate - mean velocity less than 0.1 ft./sec.) so errors in measuring the total volume delivered to the cylinder did not normally exceed $\pm 0.2\%$. That is, the mean flow rate was accurate to $\pm 0.4\%$.

Errors in the delivered solids concentration ($\pm 0.5\%$) and in the flowing concentration of solids ($\pm 1.42\%$ - using two standard deviations or 95% probability) determine the error associated with slip rate. This is exceedingly sensitive to small errors as it involves a subtraction of the flowing and delivered solids concentration and is only accurate to about $\pm 13\%$ which would explain the large scatter in slip rates. In comparing average slip rates between the two materials, the maximum estimated error of $\pm 1\%$ in calibrating the metering cylinder would result in an error of about $\pm 6.5\%$.

Solids flow rates depend on the accuracy of determining the delivered solids concentration and the solids concentration in the line and hence would have a maximum variation of $\pm 1.9\%$ for any solids material.

7. RESULTS.

7.1. Pressure drop over a straight vertically rising pipe section.

The first two graphs (37 and 38) show the pressure drop produced by water only as a function of the mean velocity for the $\frac{1}{2}$ inch and $\frac{3}{4}$ inch lines. The measurements of pressure drop and velocity for water only in the test line were converted to friction factors and Reynolds numbers and compared with standard plots of these groups. (60). The results were fairly consistent indicating a roughness to diameter ratio of 0.0008 for the $\frac{1}{2}$ inch line and 0.0002 for the $\frac{3}{4}$ inch line. These values are equivalent to 10 and 4 microns, respectively. This is rather less than the 50 to 100 micron tolerance on roughness quoted by the manufacturers but seems to agree with microscopic observation of the surface.

The dynamic pressure drop of silver sand is also plotted on these graphs as a function of mean velocity of water and sand. It will be observed that at mean velocities between about 0.3 feet/sec. and about 3.0 feet/sec. the dynamic pressure lay on or close to the pressure drop of water only. Below approximately 0.3 feet/sec. there was a tendency for the pressure drop to fall away below that attributable to static head.

Above mean velocities of about 3.0 feet/sec., the

Fig. 37.

Effect of mean velocity on the dynamic pressure drop with vertically rising flow in the $\frac{1}{2}$ inch pipe with ballotini and with water only

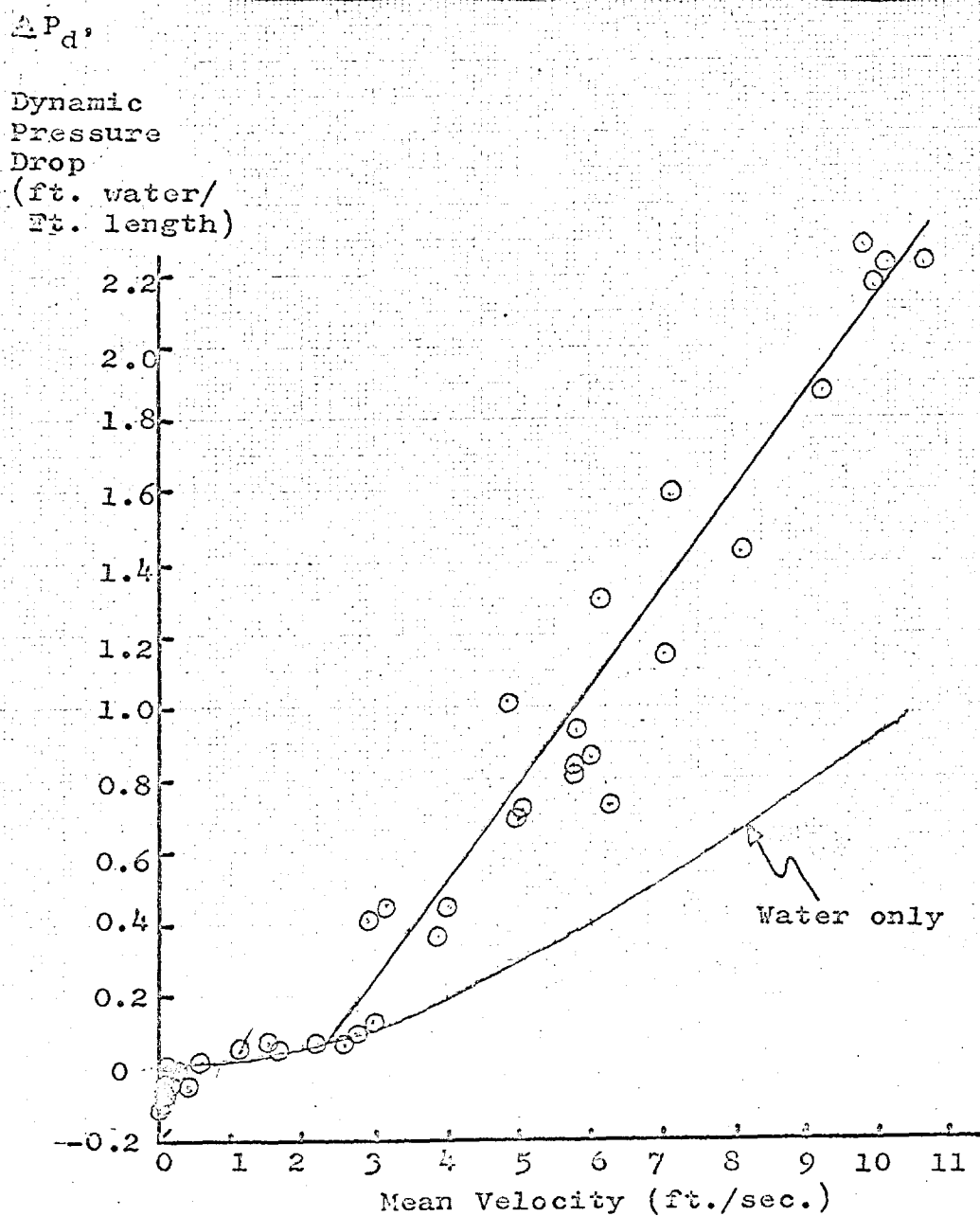
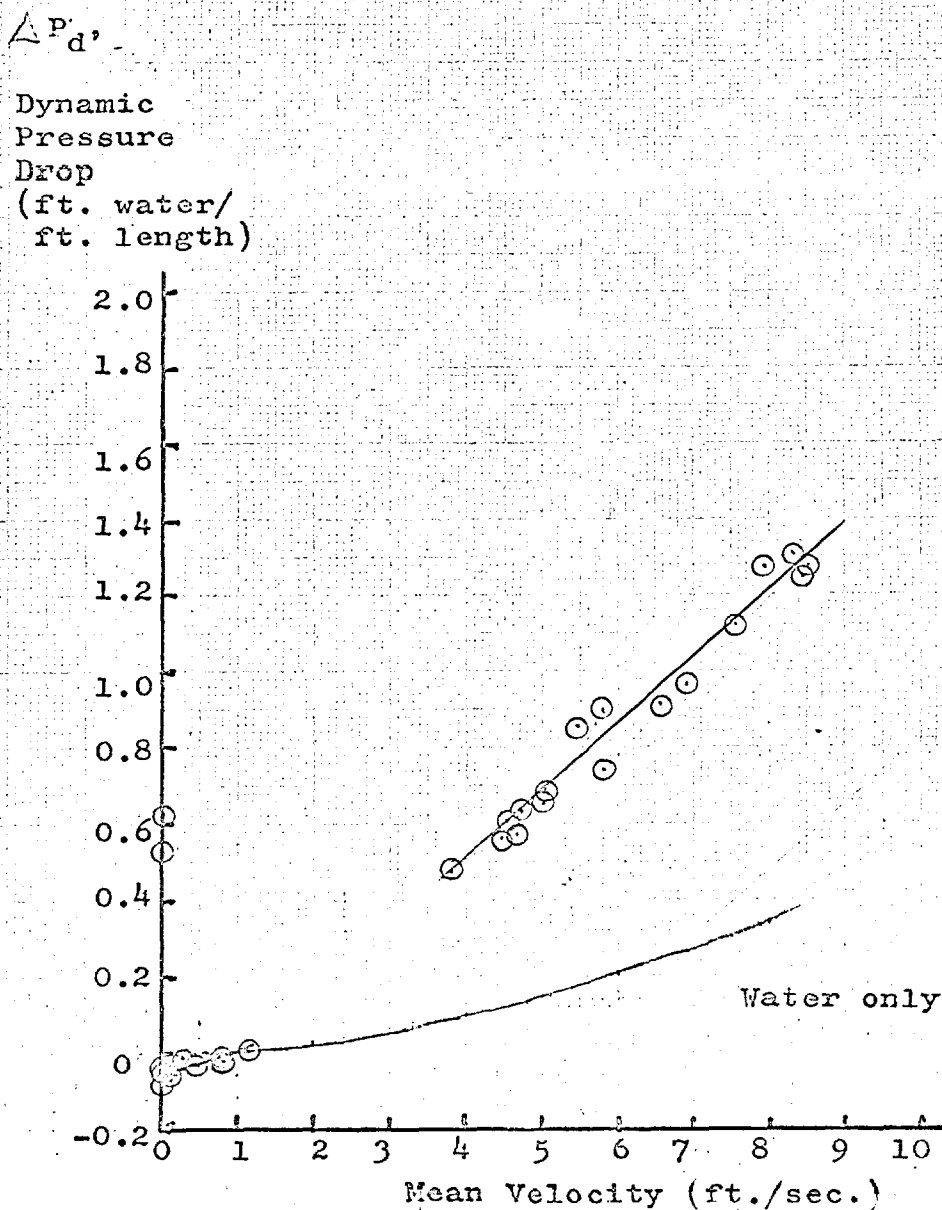


Fig. 38.

Effect of mean velocity on the dynamic pressure drop with vertically rising flow in the $\frac{3}{4}$ inch pipe with ballotini and with water only



dynamic pressure drop left the water pressure drop curve and increased linearly with increasing mean velocity. As this was completely unlike a pressure drop plot arising from the flow of fluids, a plot was also prepared of the dynamic pressure drop against the mean solids velocity. Despite the lower accuracy in determining the mean solids velocity as opposed to the mean velocity, a sample regression analysis on the results for sand and in the $\frac{1}{2}$ inch line with solids velocities greater than 5 feet/sec. (a manifestly linear section for both axes of velocity) showed a much lower standard deviation of pressure readings. When plotting against mean velocity, the standard deviation of pressure readings from a straight line of best fit was 0.093 feet water head/feet pipe length while the plot against solids velocity gave a standard deviation of 0.067 of the same units. (Fig. 39). This confirmed visual observation that in all cases plotting of solids velocity gave less scatter about a straight line. Plots of dynamic pressure drop against solids velocities are shown in fig. 40 - 43. The transition from following the water pressure curve occurs about 2 feet/sec. in terms of solids velocities. It will be noted that the two plots for ballotini behave in the same way as silver sand at rates below this

Fig. 39.

Comparison of the correlation of dynamic pressure drop with vertically rising flow in the $\frac{1}{2}$ inch pipe with sand as a function of mean velocity and of solids velocity

Dynamic
Pressure
Drop
(ft. water/
ft. length)

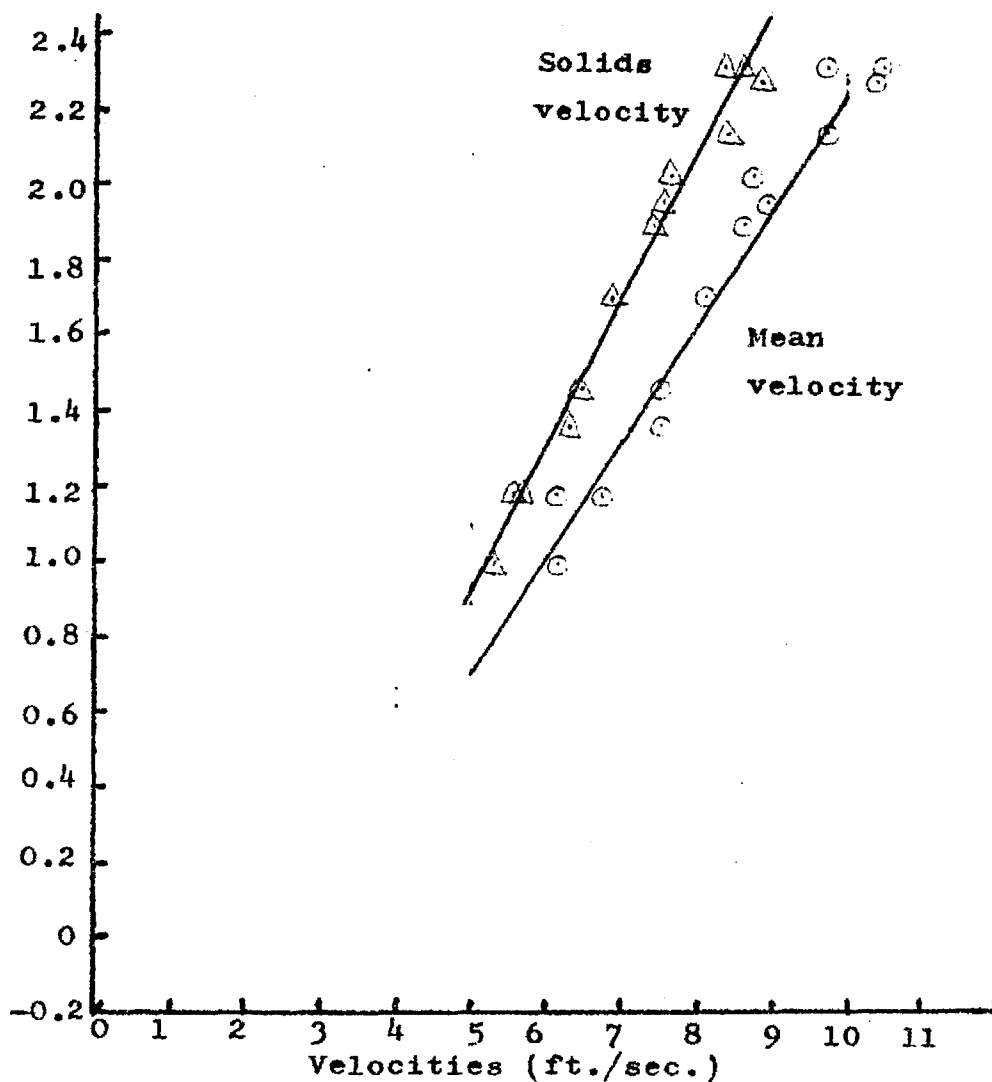


Fig. 40.

Effect of solids velocity on the dynamic pressure drop with vertically rising flow in the $\frac{1}{2}$ inch pipe with sand

ΔP_d
Dynamic
Pressure
Drop
(ft. water/
ft. length)

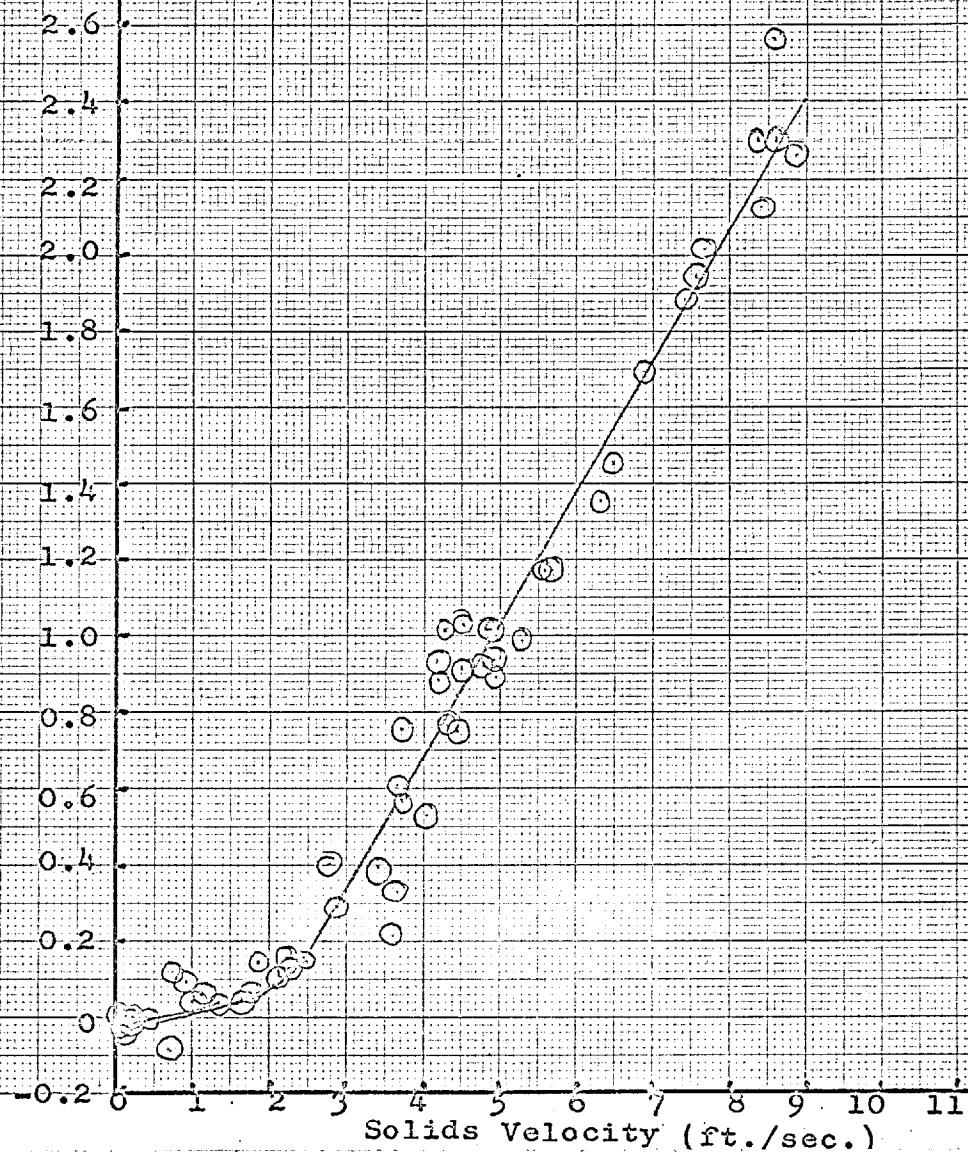


Fig. 41.

Effect of solids velocity on the dynamic pressure drop with vertically rising flow in the $\frac{1}{2}$ inch pipe with ballotini

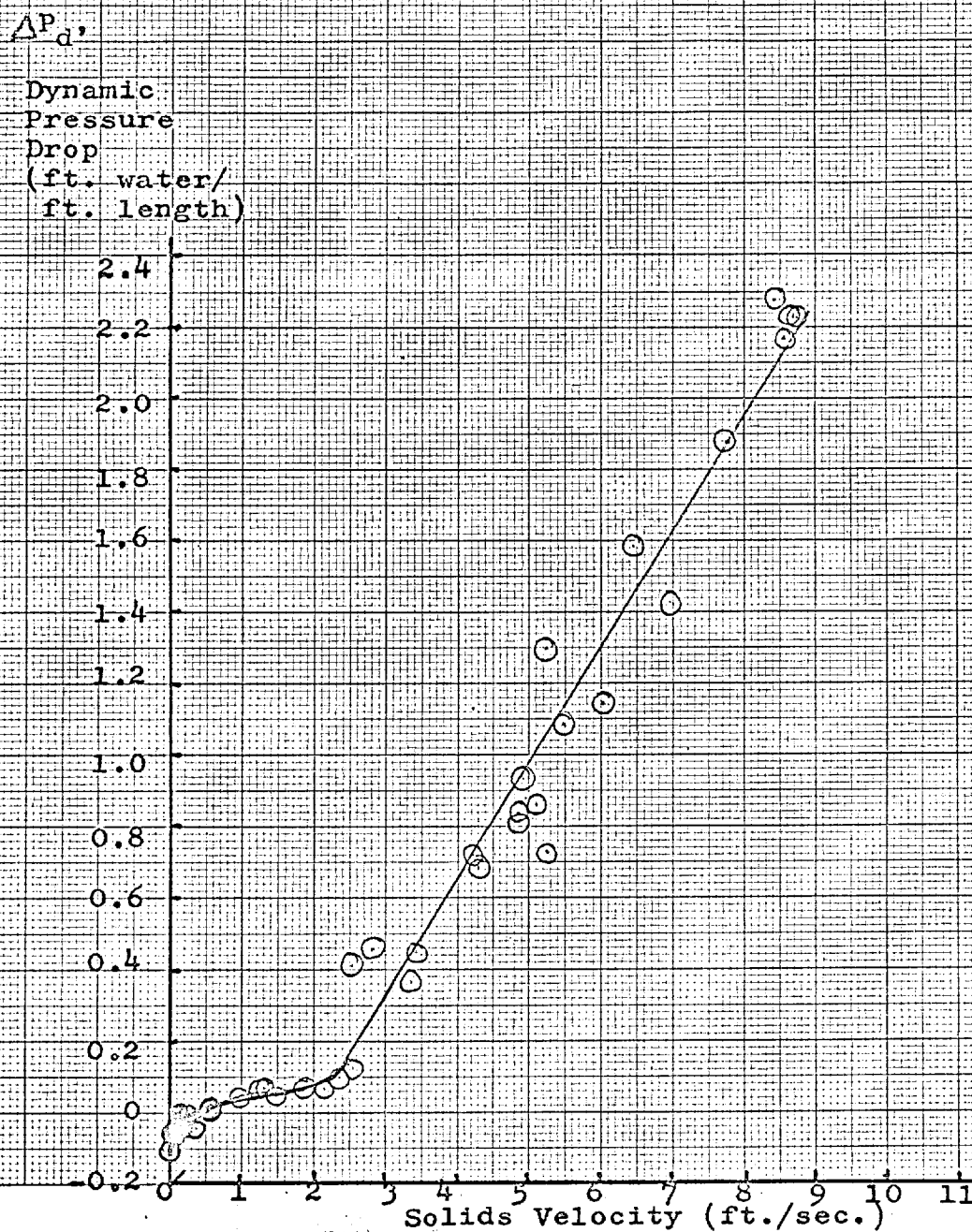


Fig. 42.

Effect of solids velocity on the dynamic pressure drop with vertically rising flow in the $\frac{3}{4}$ inch pipe with sand

ΔP_d
Dynamic
Pressure
Drop
(ft. water/
ft. length)

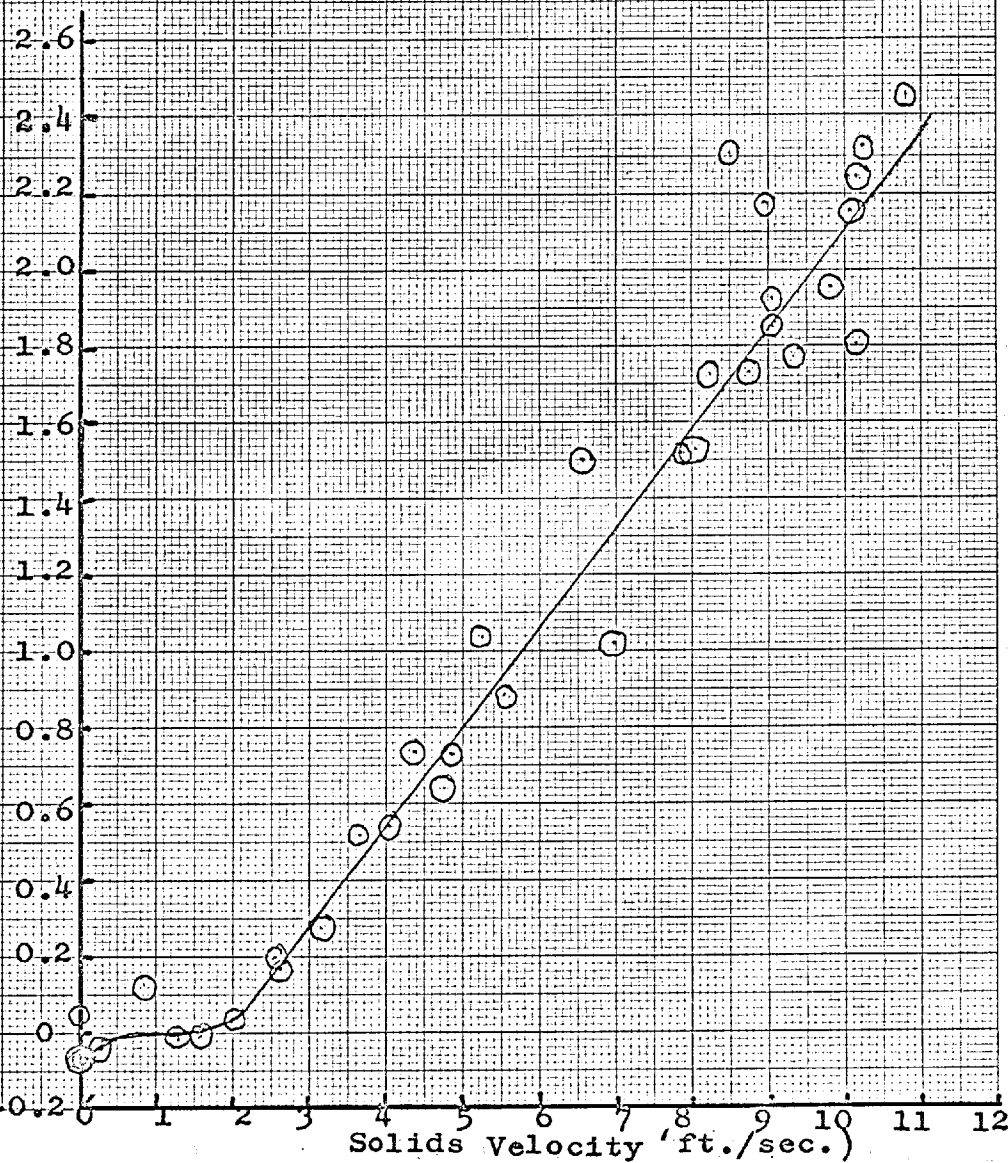
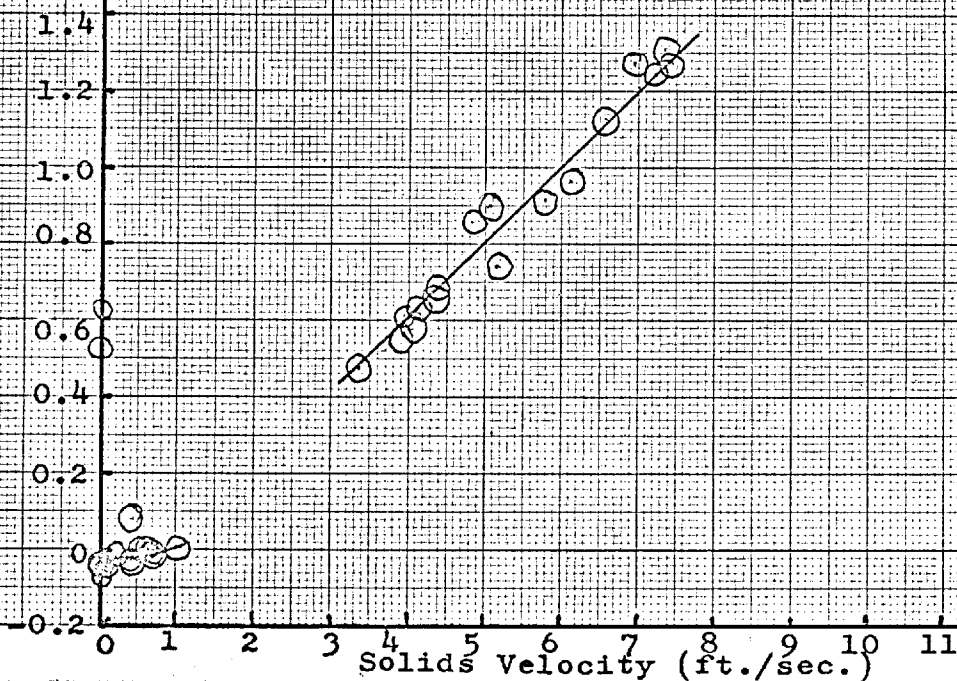


Fig. 43.

Effect of solids velocity on the dynamic pressure drop with vertically rising flow in the $\frac{3}{4}$ inch pipe with ballotini

ΔP_d
Dynamic
Pressure
Drop
(ft. water/
ft. length)



transition. The gap in the results for ballotini in the $\frac{3}{4}$ inch line was caused by an exceptionally high pressure over the whole apparatus (Section 7.3). Since similar smaller peaks in this overall pressure drop against solids velocity plot occurred in the other three cases without detectable effect on the pressure drop over the straight section of the vertically rising flow it seemed very likely that the missing section of the plot exhibited the same undisturbed characteristics observed in the other three instances.

In this section of linear increase in the pressure drop with solids velocity, there appeared to be small but sometimes marked discontinuities at about 5 feet/sec.

From the linear nature of the dynamic pressure drop velocity relationship above the 2 feet/sec. transition, it would appear that liquid friction contributed negligibly to the measured pressure drop. If liquid friction had continued above the transition the pressure drop would have been expected to include a term which varied with about the 1.8 th power of velocity.

While it is usual in studies of the vertical flow of liquids and slurries to subtract the static head of solids, plots were also prepared of the total pressure drop which measured the force applied by the

slip water on the solids particle assembly. (Fig. 44-47.)

The total force exerted by the liquid on the solids particles over unit length of the pipe is the product of the total pressure drop per unit length and the cross-sectional area of the tube. Considering this force as being uniformly distributed between the particles in this unit length:

$$\Delta P_t \times A_p = F_d \times N$$

where ΔP_t = total liquid pressure drop over unit length,

A_p = cross-sectional area of the pipe,

F_d = drag force on an individual particle,

N = number of particles in unit length of pipe.

$$\therefore \Delta P_t = \frac{F_d}{\left(\frac{\pi D^2}{4}\right)} \cdot \frac{(1-e_t) \cdot \pi D^2/4}{\frac{4}{3} \cdot \frac{\pi d_p^3}{8}}$$

where D = pipe diameter

d_p = particle diameter,

e_t = voidage in the line,

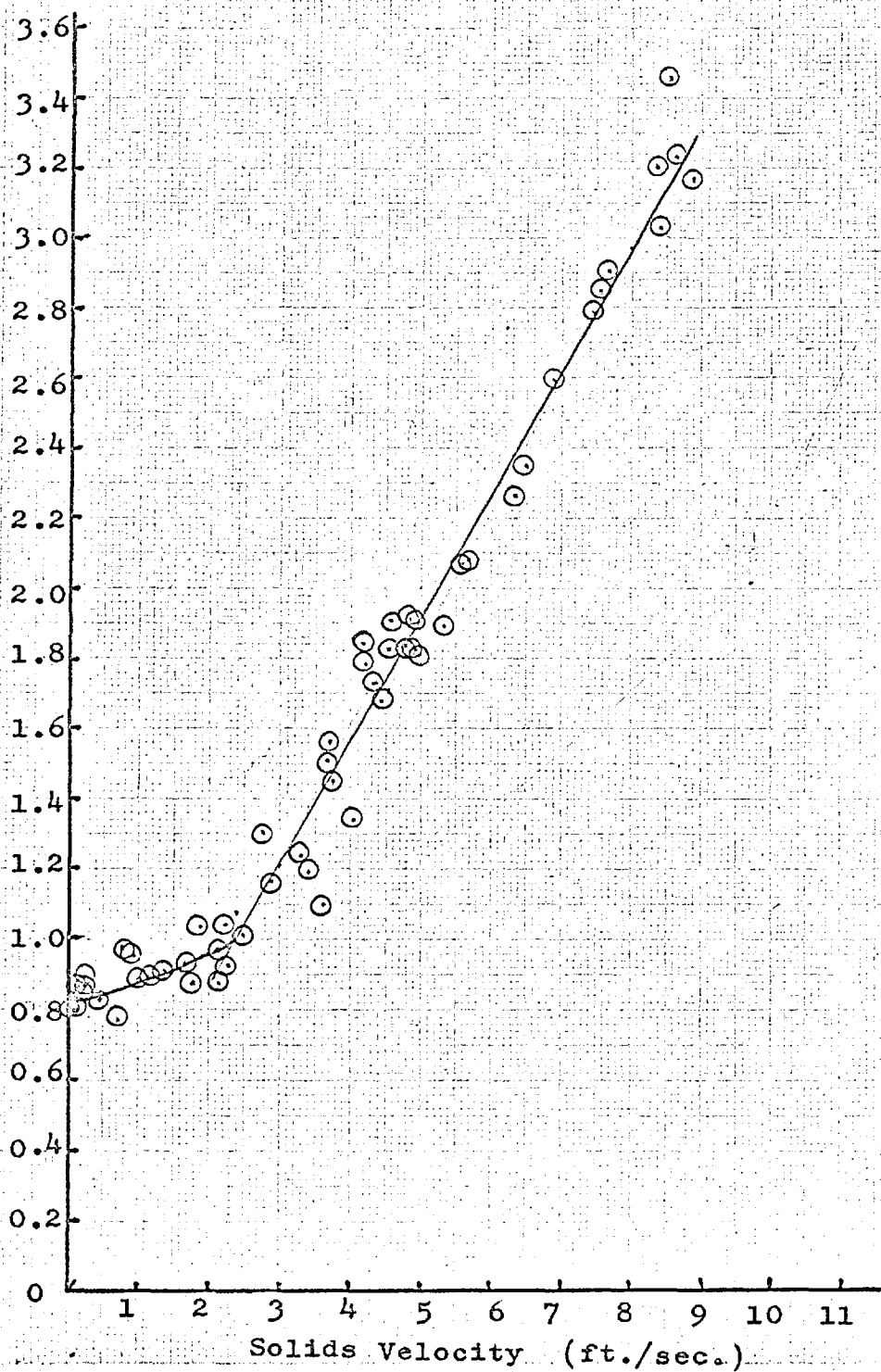
$$\therefore F_d = \Delta P_t \cdot d_p^3 \cdot \pi / (6 \times (1-e_t))$$

The drag coefficient for isolated spheres is conventionally defined (59) :

ΔP_t ,

Total
Pressure
Drop
(ft. water/
ft. length)

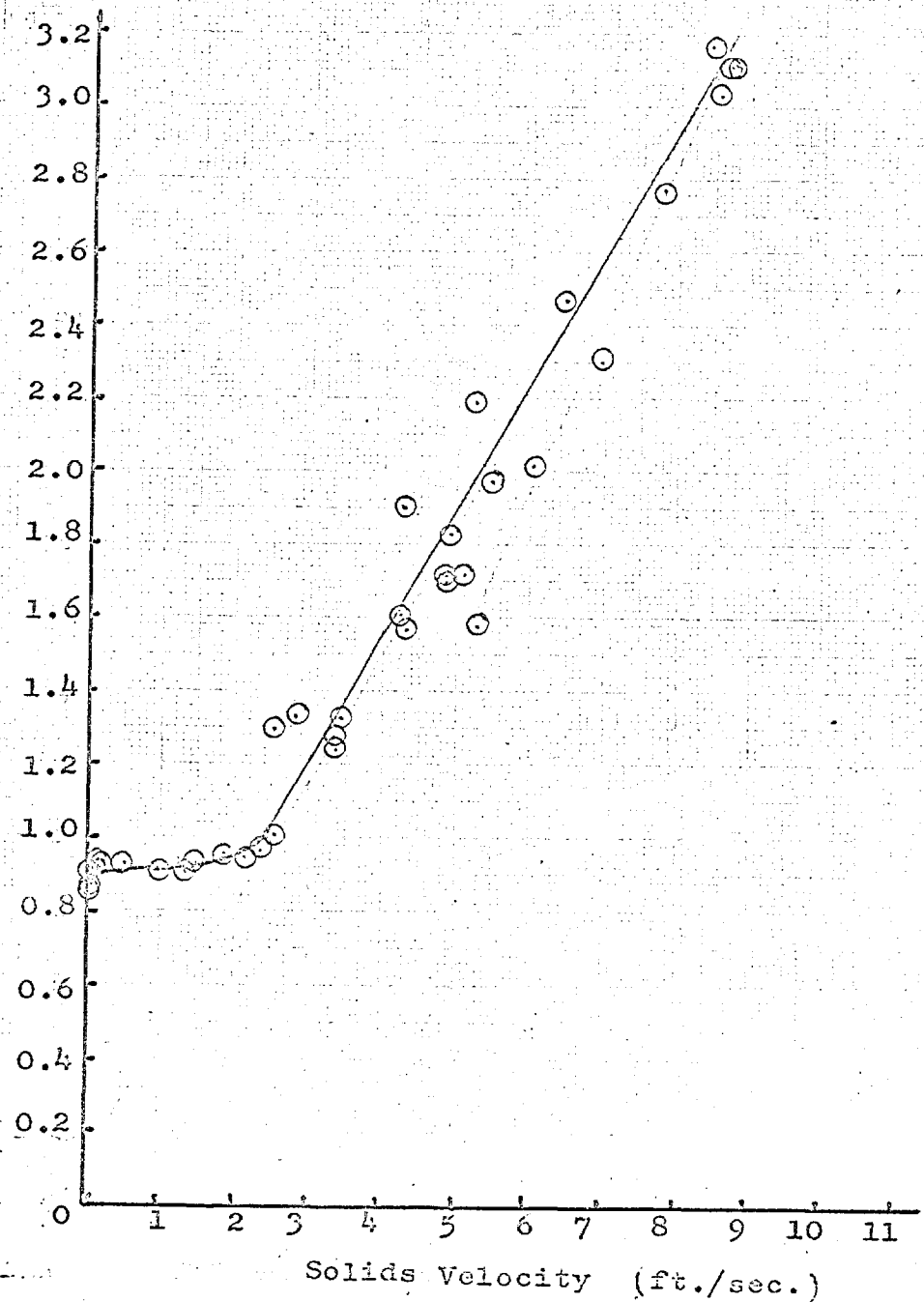
Effect of solids velocity on the total
pressure drop with vertically rising
flow in the $\frac{1}{2}$ inch pipe with sand



Effect of solids velocity on the total
pressure drop with vertically rising
flow in the $\frac{1}{2}$ inch pipe with ballotini

 ΔP_t

Total
Pressure
Drop
(ft. water/
ft. length)



Effect of solids velocity on the total
pressure drop with vertically rising
flow in the $\frac{3}{4}$ inch pipe with sand

ΔP_t ,
Total
Pressure
Drop
(ft. water/
ft. length)

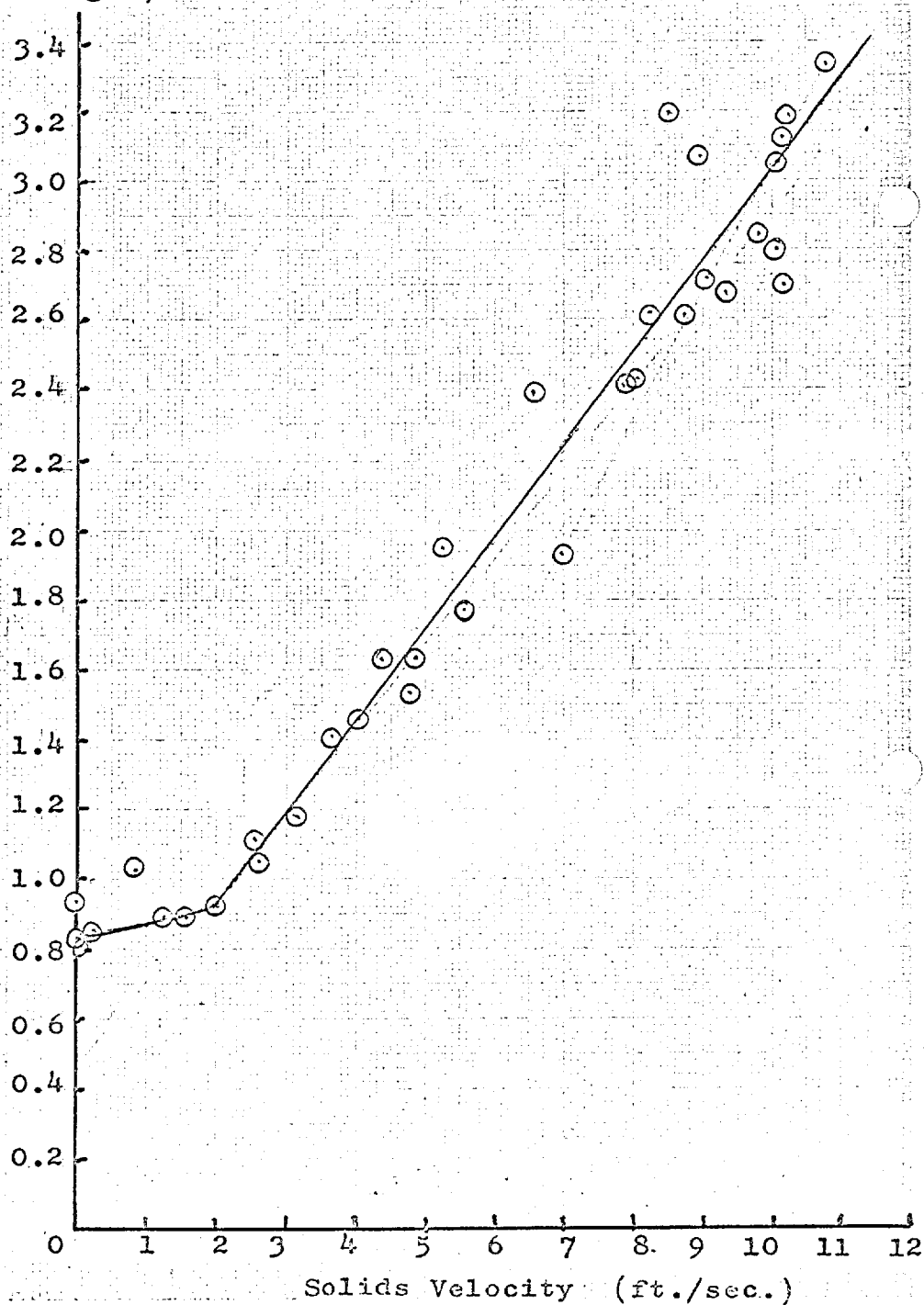
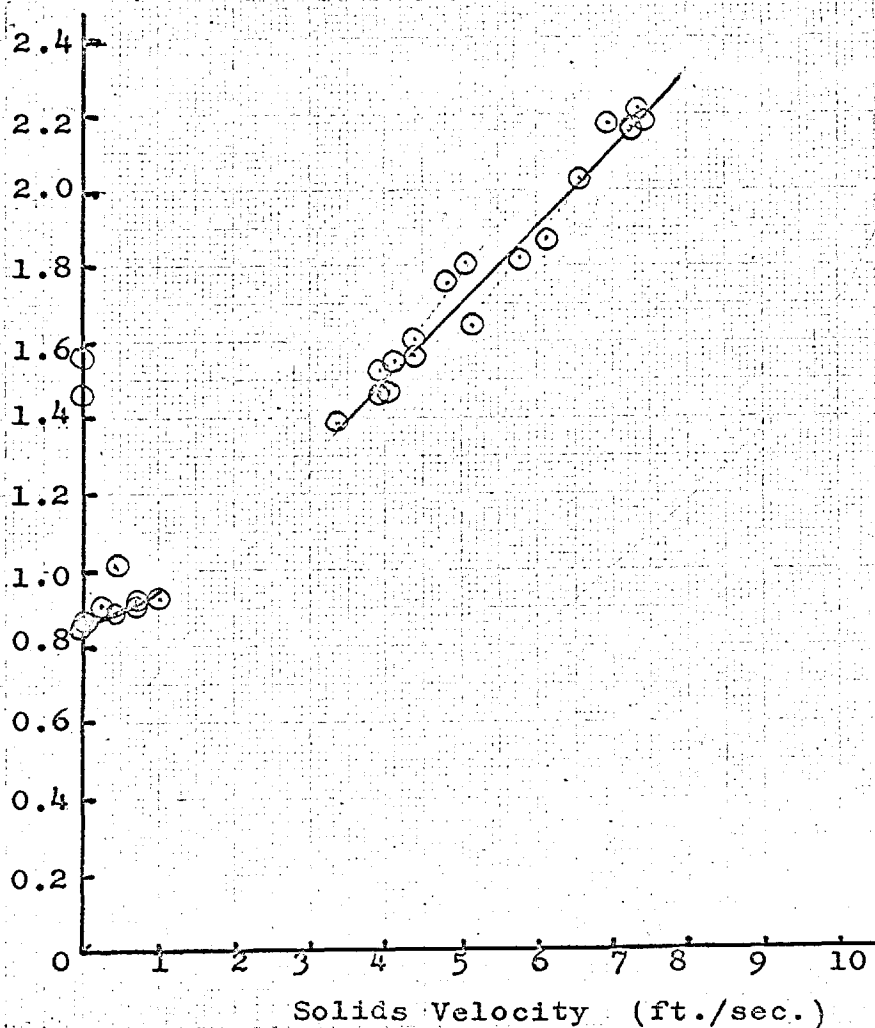


Fig. 47.

Effect of solids velocity on the total
pressure drop with vertically rising
flow in the $\frac{3}{4}$ inch pipe with ballotini

ΔP_t ,
Total
Pressure
Drop
(ft. water/
ft. length)



$$C_D = \frac{8F_d}{\pi d_p^2 \rho_1 v_{slip}^2}$$

where v_{slip} = absolute velocity of liquid past solid particle,

ρ_1 = density of the fluid.

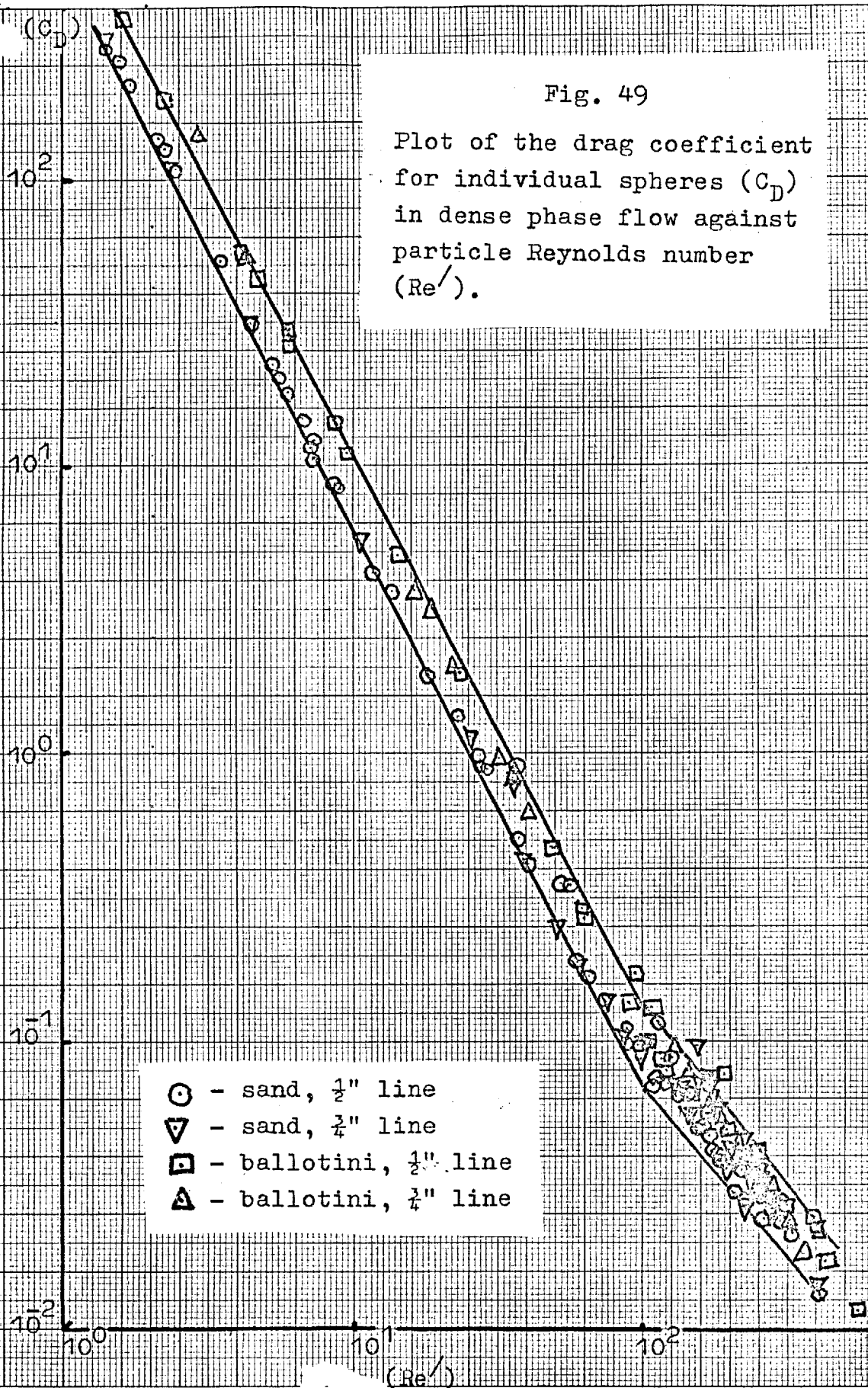
Substituting the drag force in the equation for drag coefficient,

$$C_D = \frac{4 \Delta P_t \cdot d_p}{3 \rho_1 \cdot v_{slip}^2 (1 - e_t)}$$

The drag coefficient is plotted against the particle Reynolds number, Re' in fig. 49. Re' is the conventional group using particle diameter, absolute ^{liquid} ~~solids~~ density, and slip velocity.

Fig. 49

Plot of the drag coefficient for individual spheres (C_D) in dense phase flow against particle Reynolds number (Re).



7.2. Pressure drop at right angled bends.

P3, the gauge pressure at the lower of the two tapping points, PI_1 , on the vertically rising straight section was measured. This pressure gauge measured the pressure drop occurring in a vertically rising section, a sharp right angled bend, and a short horizontal section followed by a short shallow curve through about 30° .

Nothing ~~was~~ known of the pressure drop occurring in this short horizontal section and in the shallow curve and they were assumed to be fairly small compared to the pressure drop at the sharp bend and were neglected. The pressure drop which arose in the vertically rising section was known and was subtracted from P3 leaving a pressure drop produced mainly at the right angled bend.

To check the assumption of neglecting the unknown pressure drops, the pressure drop occurring over the entire system was similarly treated by subtracting the pressure drop arising in the vertically rising sections and dividing the residual pressure drop by the total number of right angles in the system. (Five in the $\frac{3}{4}$ inch line treating the "T" below the pressure-storage vessel as a right angled bend, and four in the

$\frac{1}{2}$ inch line where flow passed straight through the "T" junction and the pressure drop caused by a sweeping 90° bend below the feed vessel was ignored.)

The results of these calculations are plotted in graphs 50 - 53. The pressure drop at a right angle is expressed as the number of pipe diameters giving the same total pressure drop in straight vertically rising flow. Neither the results from the first 53 runs - where there was a sweeping bend at the top of the vertically rising section - nor pressure drops derived from elevated overall pressure drops at low flow rates (Section 7.3) are plotted. The results based on one bend are marked Q and from bends throughout the test line K.

The plots showed a certain amount of scatter but a marked tendency to linear increase of pressure drop with increasing solids velocity. Results from the two sources of pressure measurement were in very reasonable agreement in view of the assumptions made in the calculations. The two plots for ballotini and the plot for sand in the $\frac{1}{2}$ inch line were almost concurrent when expressed in this form. The pressure drop at right angled bends for sand in the $\frac{3}{4}$ inch line, while being of the same form, is throughout rather less than half that of the other cases.

Plots were also prepared expressing the pressure drop at a right angled bend in terms of pipe diameters of dynamic pressure drop in a vertically rising section. Graph 54 is typical of the results produced thus. (N.B. Diameters are plotted at half the scale used in graphs based on total pressure drop.) The correlation was decidedly inferior to those expressed in terms of total pressure drop.

7.3. Pressure drop over the entire test line.

The pressure drop across the entire test line - the overall pressure drop (ODP) - is plotted in fig. 55 - 58. Log-log form is used to show the results at low velocity clearly, and the static head of water only is subtracted from the indicated pressure. (Section 6.4).

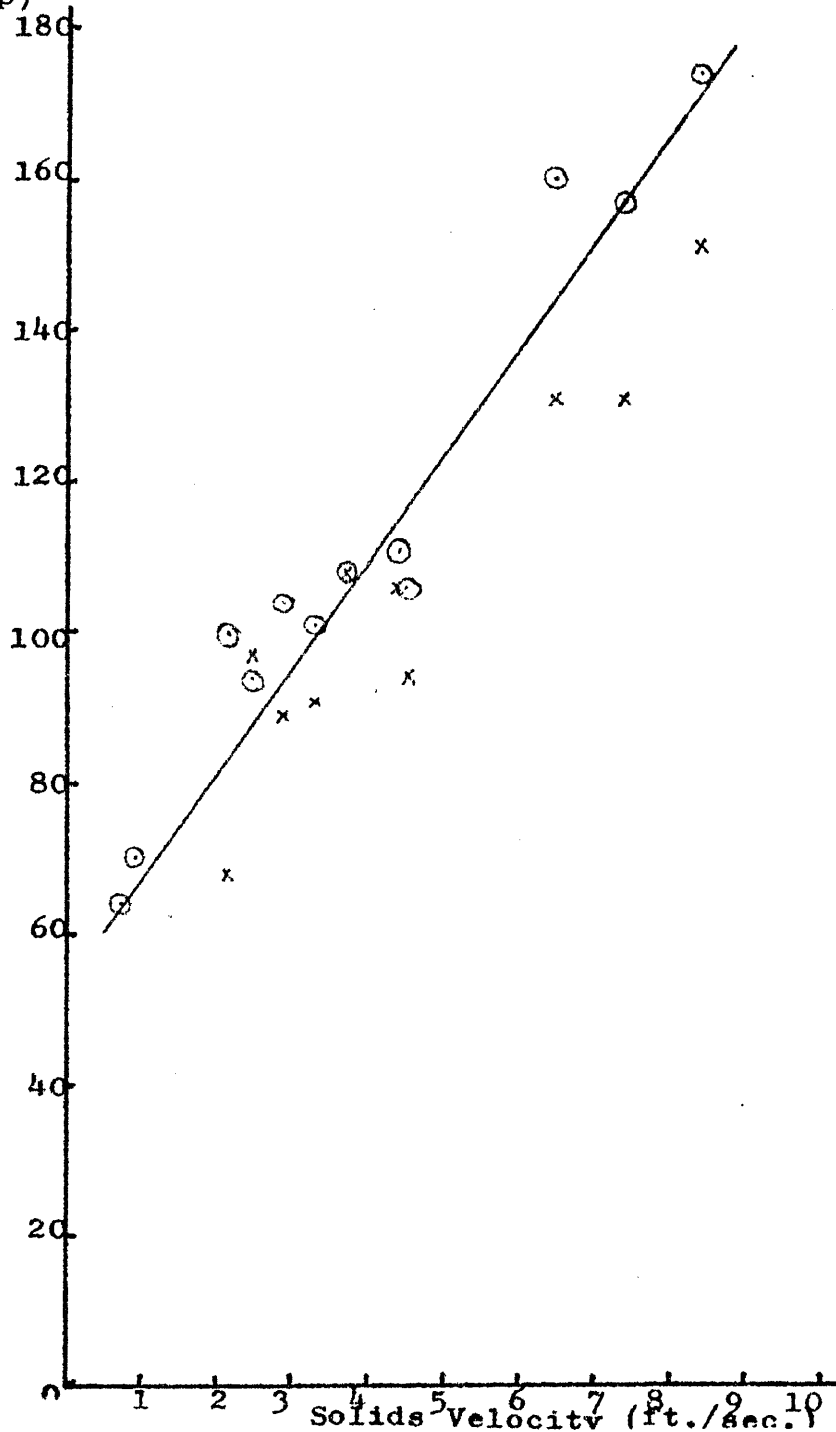
Since the overall pressure drop occurred over a very complex pipe system, the main features of interest were the transitions in the pressure drop versus flow rate pattern. A transition at about 2 to 2.5 ft./sec. solids velocity was present in all cases corresponding to the most marked transition in the pressure drop over a straight vertically rising section of pipe.

There was also a very marked step transition in all cases. The flow rates at which it occurred vary for different conditions from about 0.2 feet/sec. for

Fig. 50.

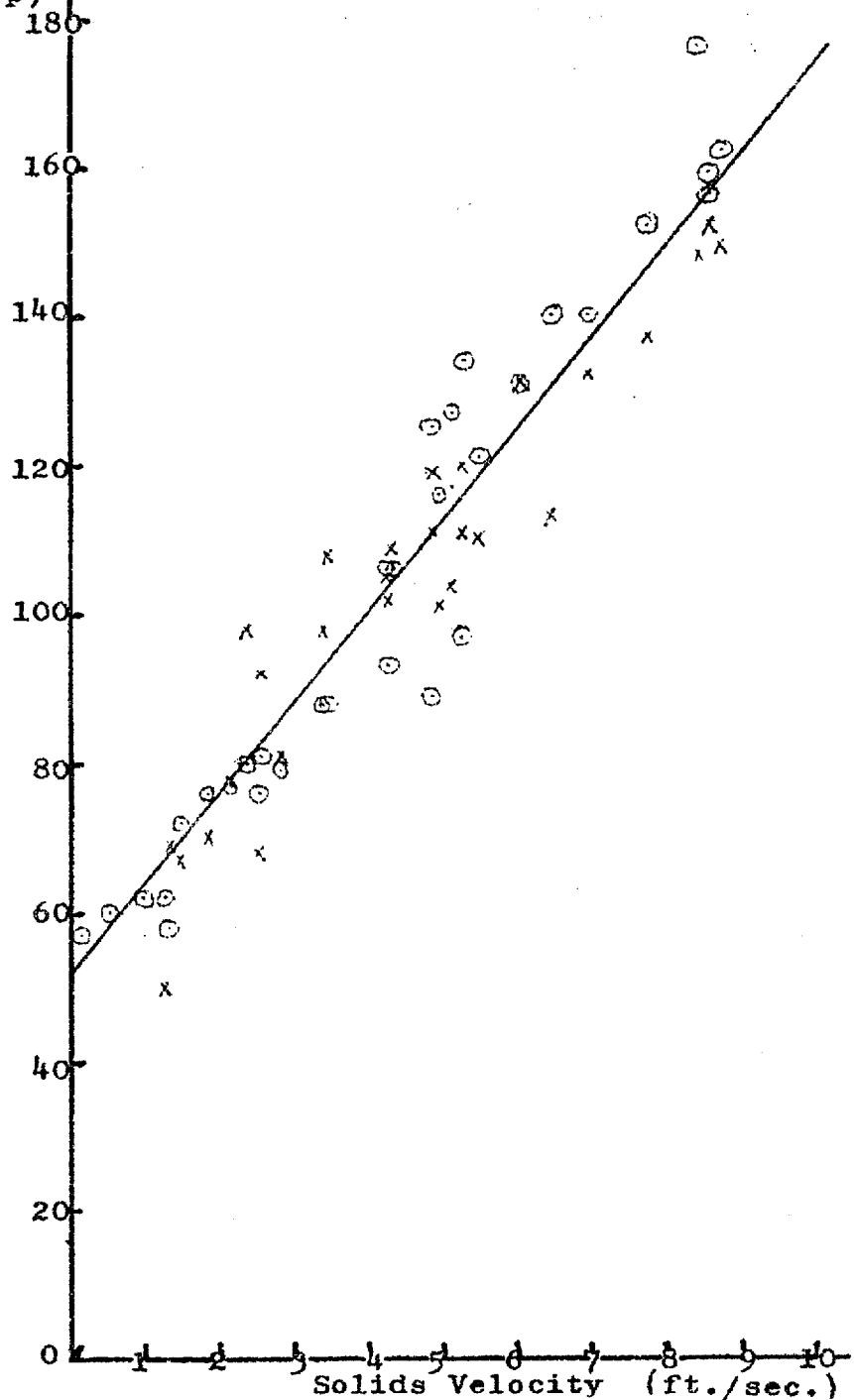
Pressure
Drop
at Right
Angled
Bend
(Pipe
diams.
of Total
Pressure
Drop)

Effect of solids velocity on the
total pressure drop at a right
angled bend in the $\frac{1}{2}$ inch pipe
with sand



Pressure
Drop
at Right
Angled
Bend
(Pipe
diam.
of Total
Pressure
Drop)

Effect of solids velocity on the
total pressure drop at a right
angled bend in the $\frac{1}{2}$ inch pipe
with ballotini



Effect of solids velocity on the
total pressure drop at a right
angled bend in the $\frac{3}{4}$ inch pipe
with sand

Pressure
Drop
at Right
Angled
Bend
(Pipe
diams.
of Total
Pressure
Drop)

120

100

80

60

40

20

0

Solids Velocity (ft./sec.)

1

2

3

4

5

6

7

8

9

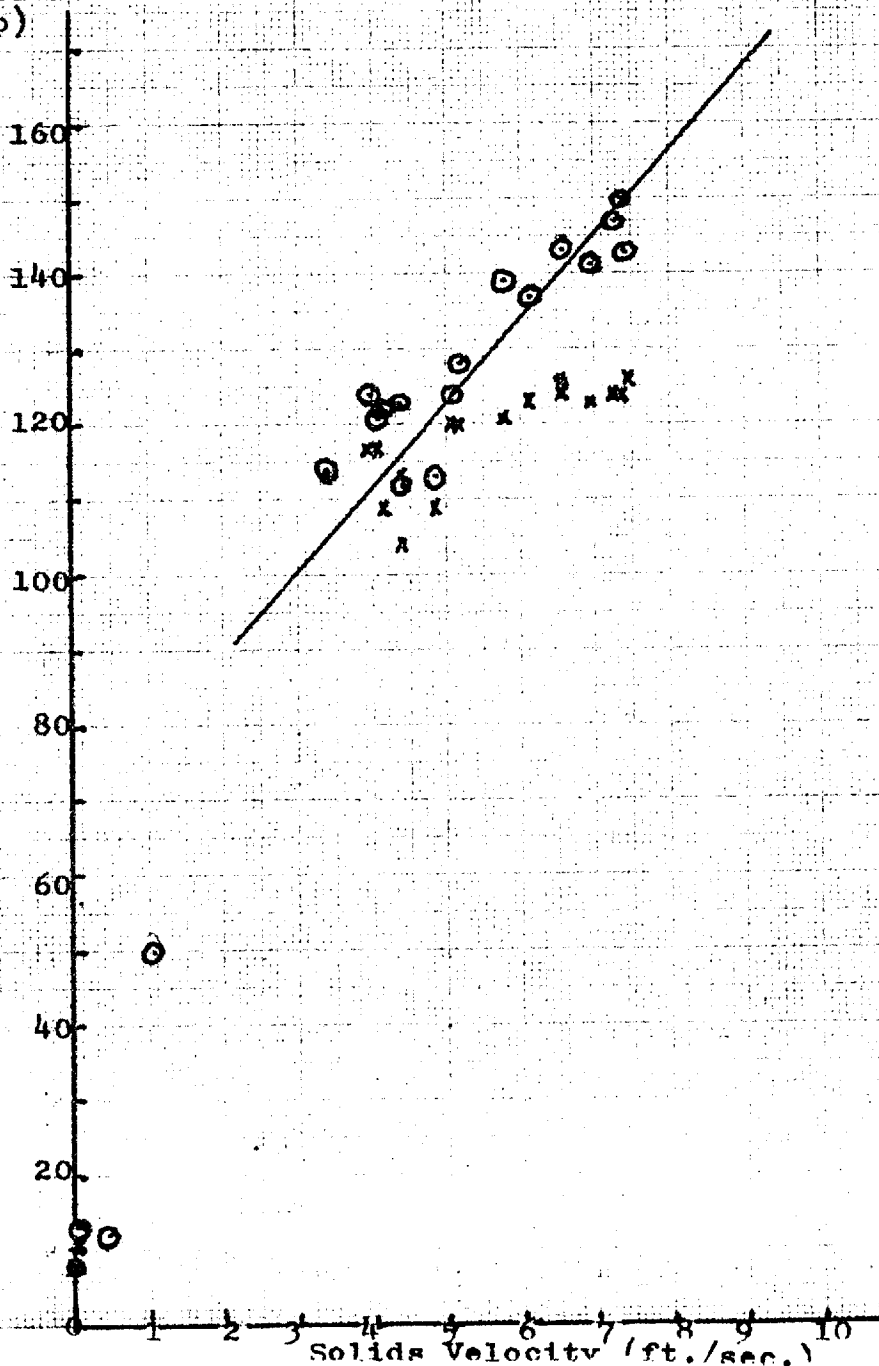
10

11

Fig. 53.

Pressure
Drop
at Right
Angled
Bend
(Pipe
diams.
of Total
Pressure
Drop)

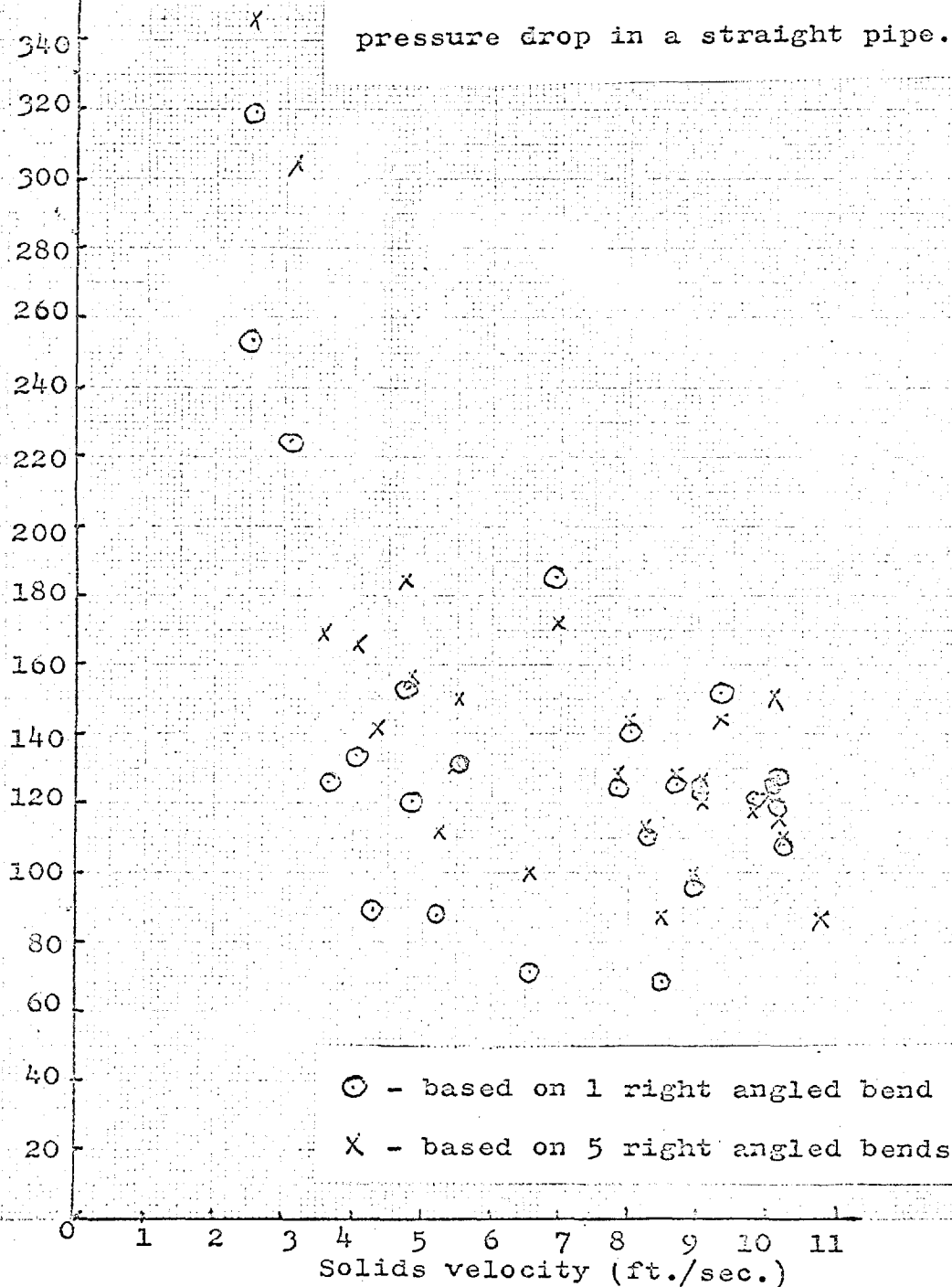
Effect of solids velocity on the
total pressure drop at a right
angled bend in the $\frac{3}{4}$ inch pipe
with ballotini



Pressure drop
at Right
Angled
Bend (Pipe
diams.
of
Dynamic
Pressure
Drop)

Fig. 54.

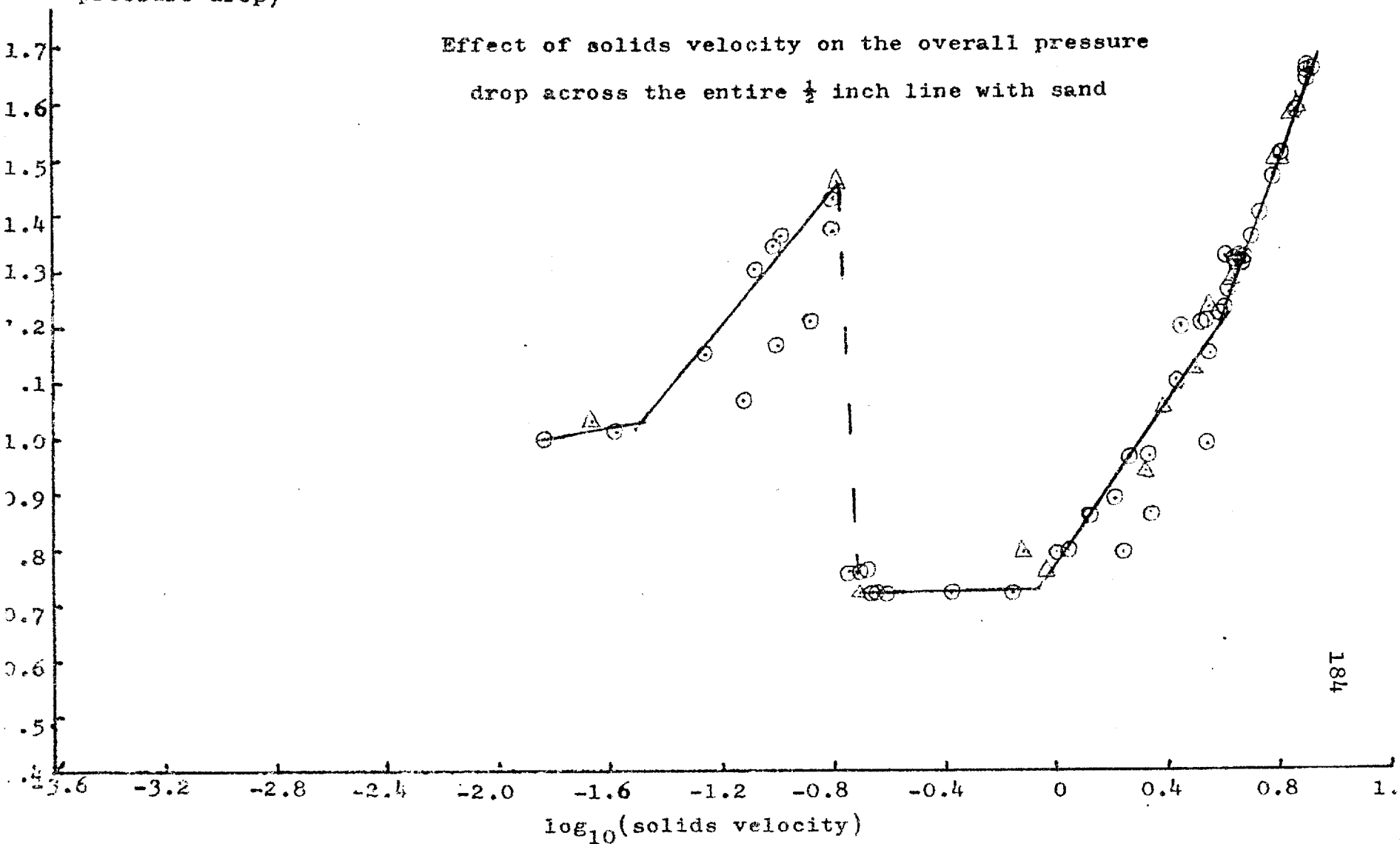
Effect of solids velocity on the total pressure drop at a right angled bend in the $\frac{3}{4}$ inch pipe with sand. The pressure drop is expressed as diameters of dynamic pressure drop in a straight pipe.



$\log_{10}$
(overall total
pressure drop)

Fig. 55.

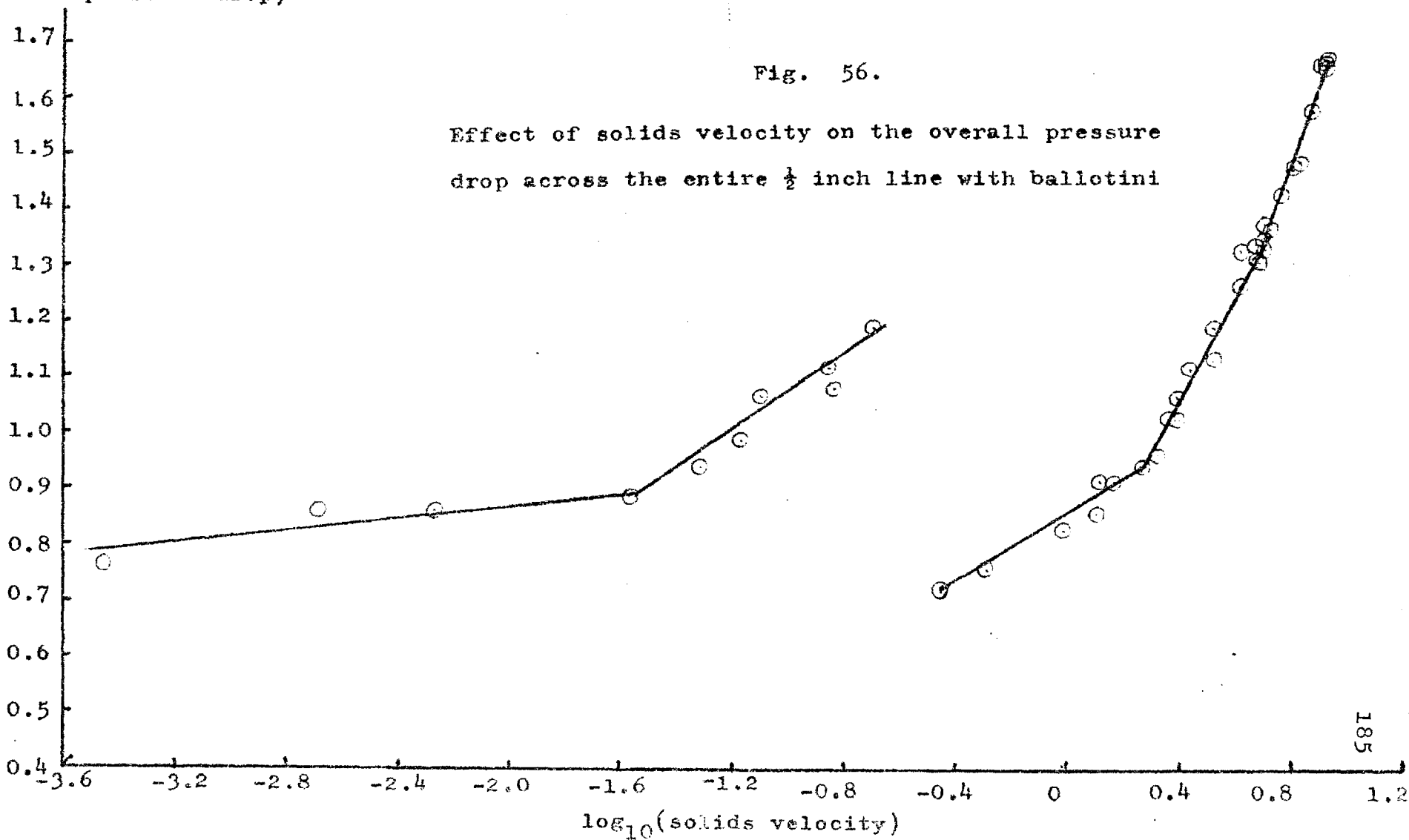
Effect of solids velocity on the overall pressure
drop across the entire $\frac{1}{2}$ inch line with sand



$\log_{10}$
(overall total
pressure drop)

Fig. 56.

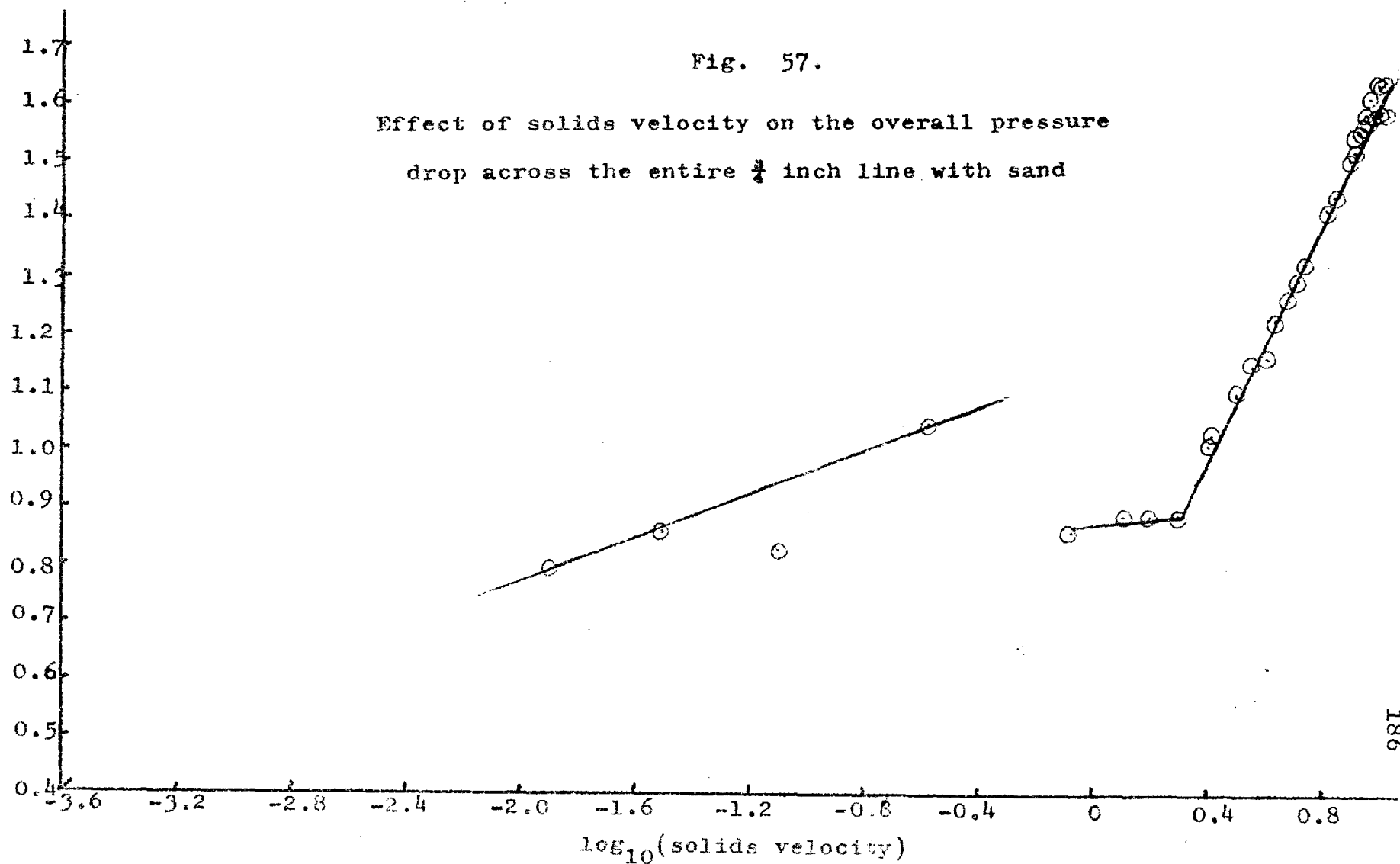
Effect of solids velocity on the overall pressure
drop across the entire $\frac{1}{2}$ inch line with ballotini



$\log_{10}$
(overall total
pressure drop)

Fig. 57.

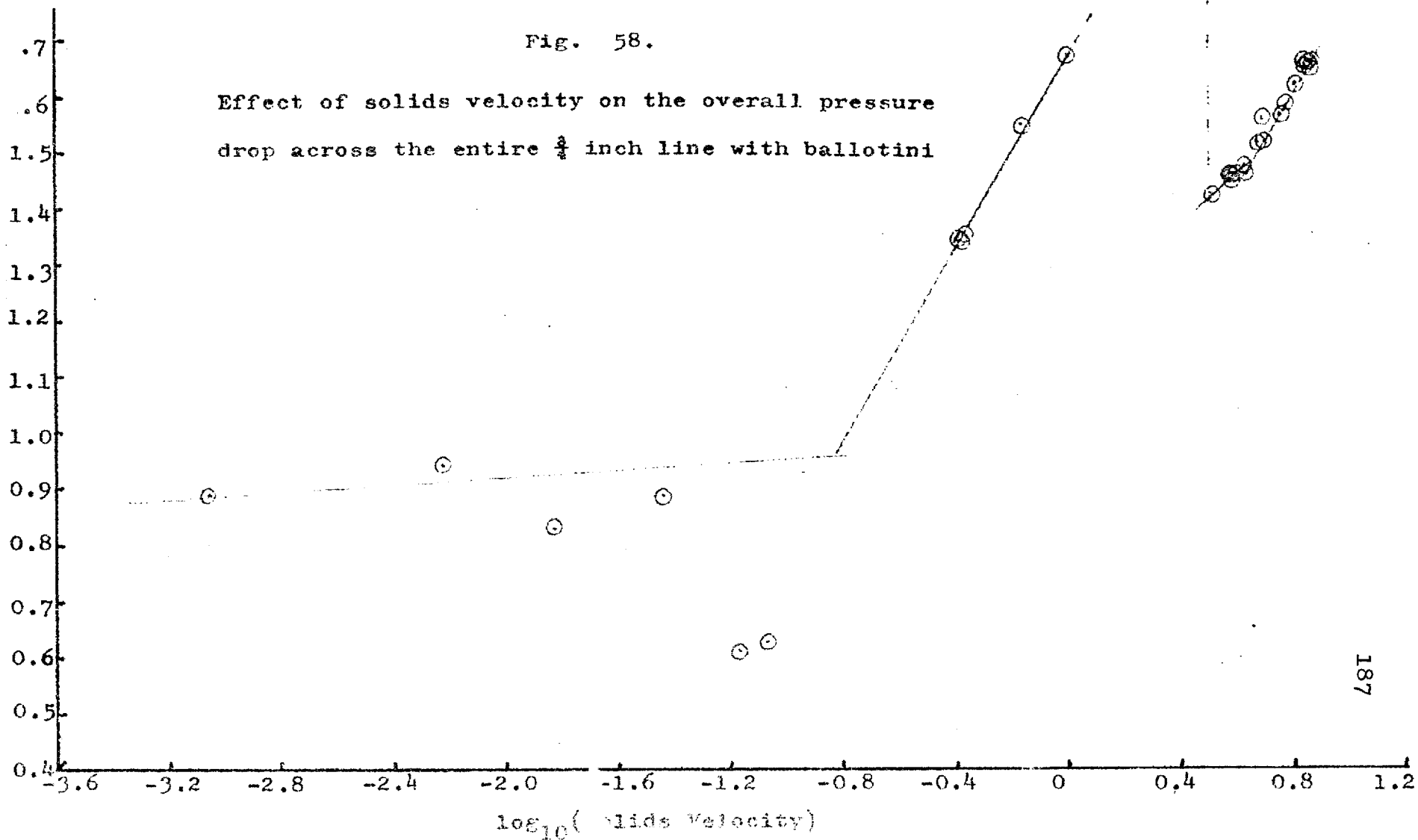
Effect of solids velocity on the overall pressure
drop across the entire $\frac{1}{4}$ inch line with sand



$\log_{10}$
(overall total
pressure drop)

Fig. 58.

Effect of solids velocity on the overall pressure
drop across the entire $\frac{3}{4}$ inch line with ballotini



sand in the $\frac{1}{2}$ inch line to nearly 4 feet/sec. for ballotini in the $\frac{3}{4}$ inch line.

Since this effect does not occur with P3, this sharp increase in pressure drop must arise between the feed vessel and the lower pressure tapping point on the vertically rising section. Since the horizontal and vertically rising parts of this section were virtually identical to those over which P3 occurred it seemed probable that the high pressure drop occurred in the vertically falling section or at the bends where horizontal flow gave way to vertically upward flow. To decide between these two possibilities, a gauge was fitted at the only readily available position. This was in the other arm of the "T" where mains water could be introduced to flush the test line of solids. At this point vertically downward flow ends and horizontal flow begins. The pressure drop here remained completely in step with the overall pressure drop. Thus the high pressure drop must be associated with the complicated section through the density gauge.

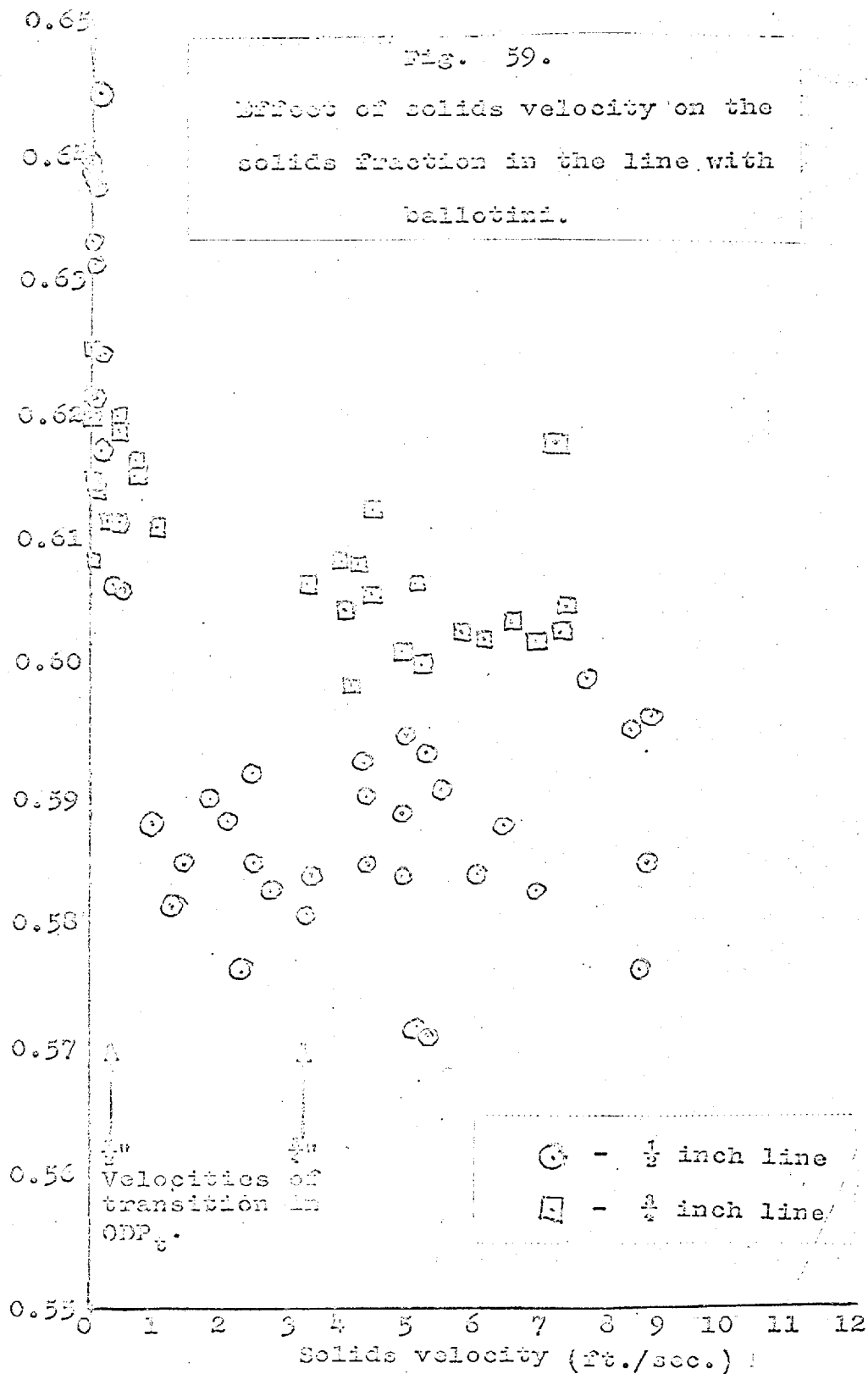
It will be noted that with ballotini in the $\frac{3}{4}$ inch test line the overall pressure drop appears to rise to about 120 lb./in². The pumps are only capable

of developing 48 lb./in^2 , but the linear nature of the log-log plot at velocities below the transition and up to 48 lb./in^2 , suggest that the extrapolation is valid. There is probably significance in the transmitted solids fraction for ballotini in the $\frac{3}{4}$ inch line where the sharp step transition is most marked. The transmitted solids fraction is very high at flow rates above this transition and even higher at rates below the transition. The ballotini in the $\frac{1}{2}$ inch line shows much the same level of transmitted solids fraction at rates below the transition but above the transition is less than that for the $\frac{3}{4}$ inch line except for a few points immediately above the transition velocity. (Fig. 59). However, this pattern is not repeated with the generally lower voidages of the silver sand.

7.4. Slip.

The slip ratios are set out in fig. 60 - 63. Slip ratios appear to be constant down to less than 1 foot/sec. and probably as low as about 0.3 feet/sec. There is, for the reasons explained in Section 6.5, a very wide scatter of individual slip ratios. The averages of slip ratios at solids velocities of greater than 1 foot/sec. for the four cases of different line

(1 - ϕ_s),
Solids
Fraction
in line.



Slip
Rate
(% of
solids
velocity)

Fig. 60

Slip rate with sand in the $\frac{1}{2}$ inch
pipe versus solids velocity.

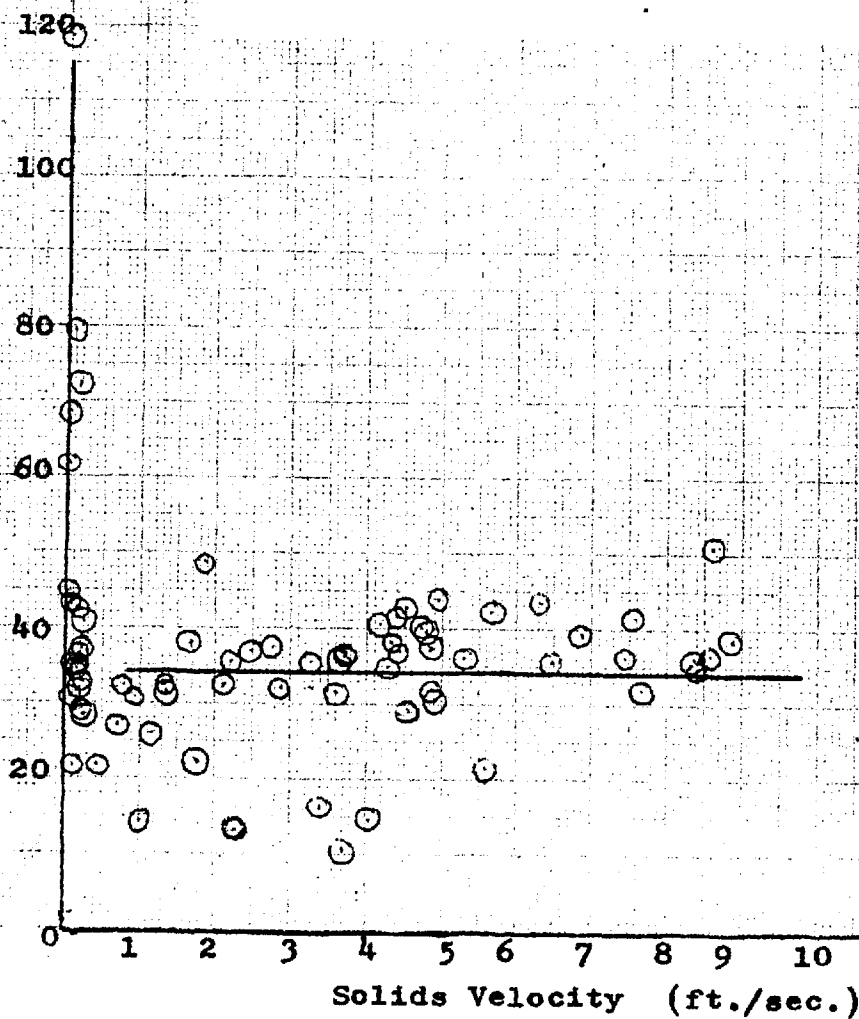


Fig. 61.

Slip rate with ballotini in the $\frac{1}{2}$ inch
pipe versus solids velocity

Slip
Rate
(% of
solids
velocity)

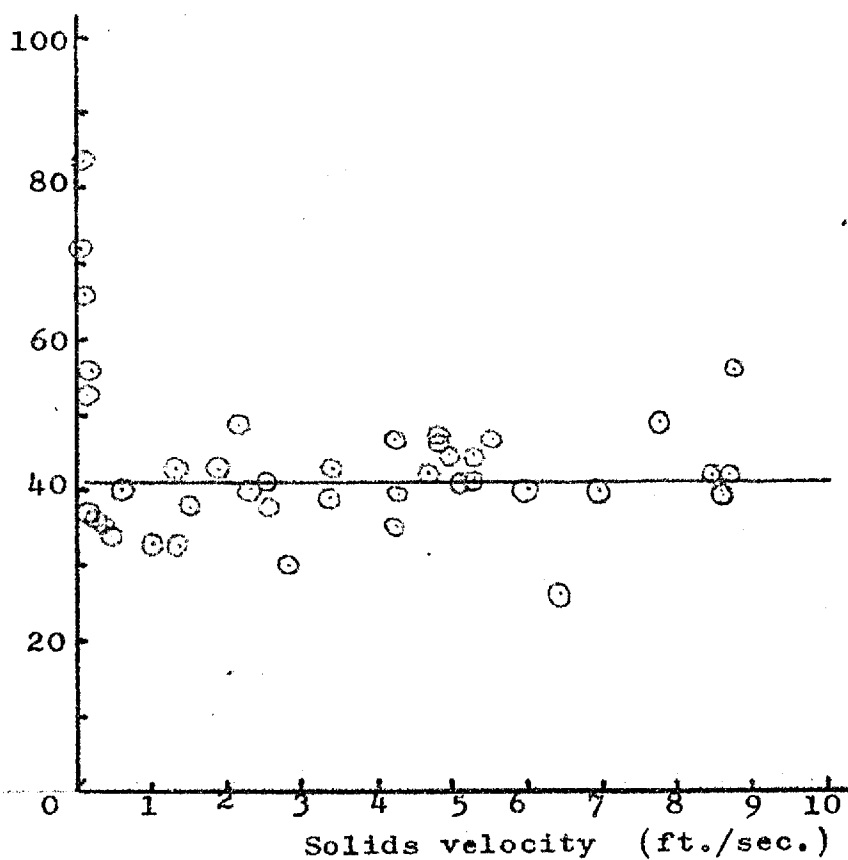
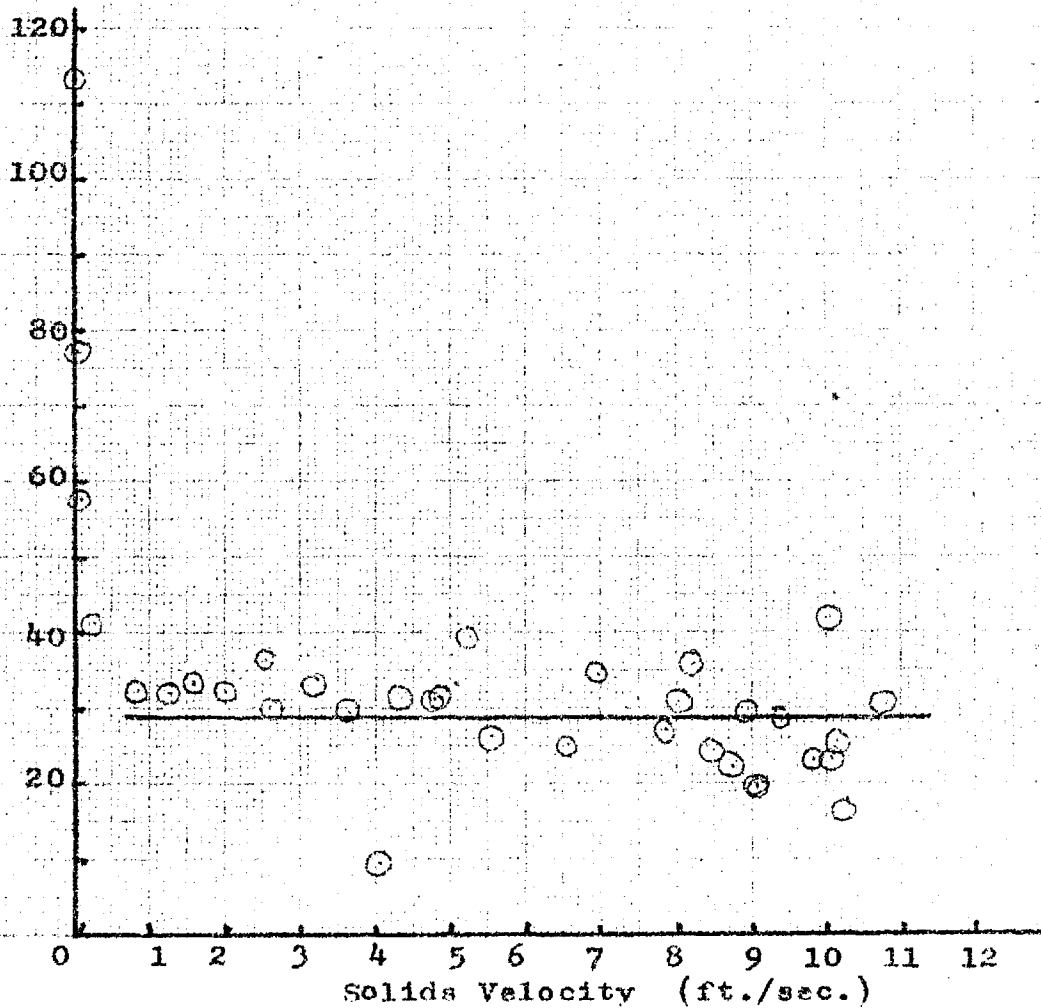


Fig. 62.

Slip ratio with sand in the $\frac{3}{4}$ inch
pipe versus solids velocity

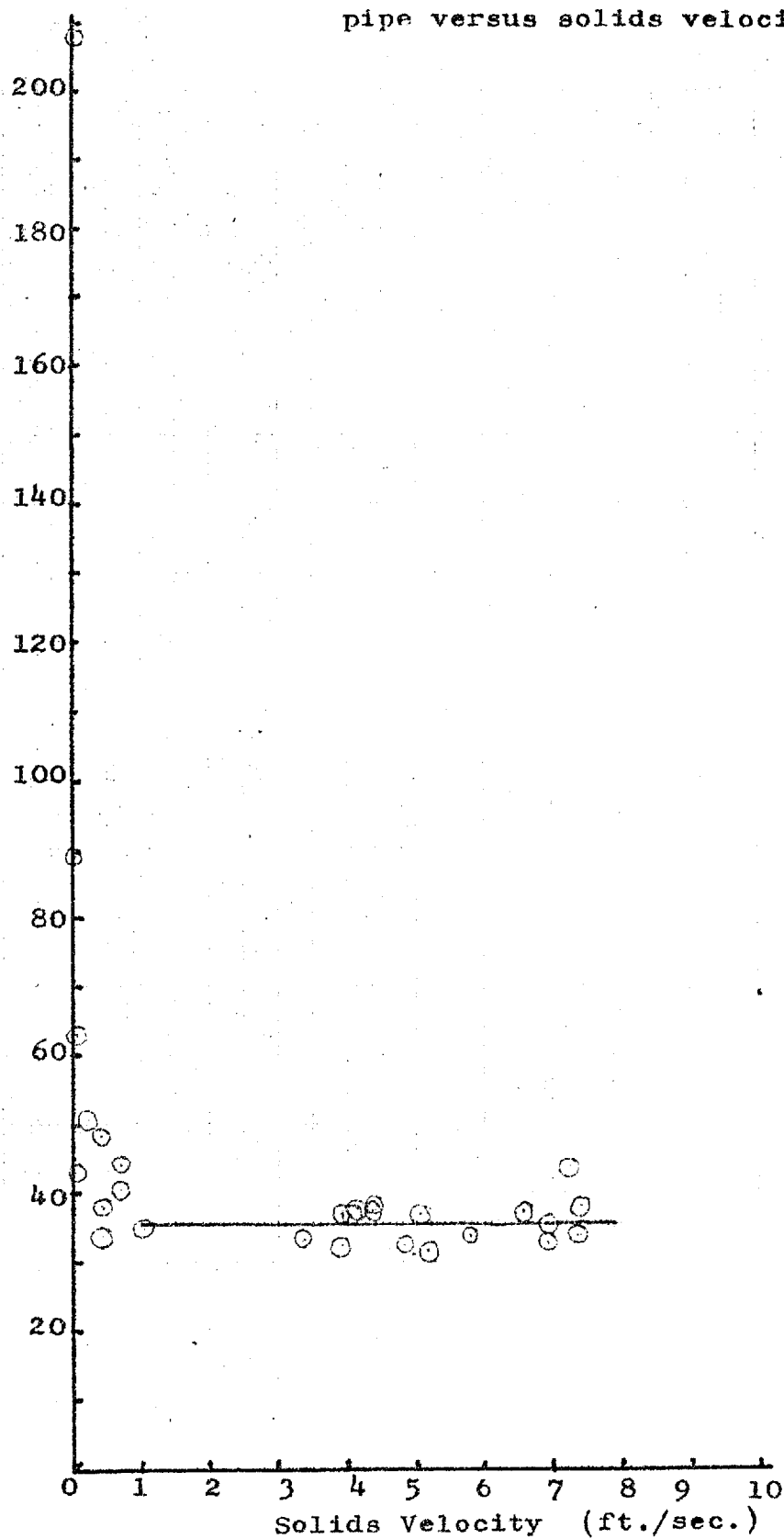
Slip
Ratio
(% of
solids
velocity)



Slip
Rate
(% of
solids
velocity)

Fig. 63.

Slip rate with ballotini in the $\frac{3}{4}$ inch
pipe versus solids velocity



diameters and materials were calculated and compared by 't' tests to see whether they belonged to different populations. These 't' tests are set out in Appendix 5. The results indicate better than 0.1% significant difference in three of the comparisons and better than 2% significance in the comparison of sand between the $\frac{1}{2}$ " and the $\frac{3}{4}$ " line. In view of possible error in measuring the slip ratios arising from calibration of the metering cylinder for the two materials (see section 6.5) the estimated maximum error was used to make the worst possible displacement of slip ratio averages towards each other. The 't' tests between materials were repeated and were still significant to better than 1% for the $\frac{3}{4}$ inch line and almost 1% for the $\frac{1}{2}$ inch line.

Below about 0.3 feet/sec. solids velocity, the slip ratio rose rapidly to reach infinity when the slip velocity was about 0.01 feet/sec. At this point and at lower velocities no solids were transferred through the line. The slip velocity at which solids transmission ended corresponded reasonably well with the measured incipient fluidization velocity.

7.5. Pressure drop across the expansion to 3 inches within the $\frac{1}{2}$ inch line.

Above solids velocities of 0.98 feet/sec., the

characteristics of the $\frac{1}{2}$ inch line with a small section replaced by the expansion to 3 inch and the subsequent contraction to $\frac{1}{2}$ inch were identical to those occurring in the simple $\frac{1}{2}$ inch line. With solids velocities of less than 0.98 feet/sec. flow of solids ceased completely and the pressure drop rose to the maximum available. Flow could not be re-started and on the first occasion that this occurred the 3 inch expansion had to be removed to extricate the solids. Adding mains water direct to the 3 inch section did not improve the situation though solids were cleared downstream from this addition, but a small water bleed immediately below the expansion cleared the blockage after a variable pause of up to a few minutes. The exact point at which the transition to jamming occurred can be pinpointed since one run jammed only moments after the results had been collected.

It seems likely that the blockage arose when the slip velocity was insufficient to maintain superficial fluidizing velocity in the expansion. The slip velocity in the run which subsequently jammed was 0.317 feet/sec. which gave an absolute average velocity in the 36 fold expansion of 0.0088 feet/sec. This was in reasonable agreement with the measured minimum absolute velocity

of fluidization for the ballotini of 0.0080 feet/sec. in view of the limited accuracy of slip ratio measurements.

It was noted in section 2.3.2. that in the pilot plant with its 1 inch to 6 inch expansion that flow rates of about 23 l./min. gave rise to jamming. The absolute slip water velocity in the separator at 30.3 l./min. was estimated to be 0.0144 feet/sec. which would indicate an absolute slip velocity of 0.0108 feet/sec. at 23 l./min. The measured absolute velocity of minimum fluidization for silver sand was 0.0087 feet/sec. indicating that the jamming observed was of similar nature.

7.6. Comparison of work to lift solids in dense phase and dilute phase.

Since the flow rate to convey the same quantity of solids through a given pipe must be considerably higher in dilute phase than in dense phase, the comparison between the two states is quite complicated. According to Durand (49) and Worster (42), the pressure drop (less the static head associated with lifting the solids) is the same as with water flowing at the same mean velocity. The solids velocity is however, rather

less than the mean velocity with the slip velocity reported as being equal to the free-settling velocity of the solids particles. On this basis the dilute phase velocity to convey the same volume of solids as in dense phase is given by:

$$V_{dil} = (V_s(1-e_t)/(1-e_{dil})) + V_p \cdot e_{dil}$$

where V_{dil} = dilute phase ^{average} ~~liquid~~ velocity,

V_s = dense phase solids velocity,

V_p = free-fall particle velocity,

e_{dil} = voidage of dilute phase flow.

Worster (~~62~~) considers that about 80 percent of the potential energy of the conveying water could normally be recovered and the ratio of work done in dilute phase and dense phase conveying is given by:

$$\frac{W_{dil}}{W_{den}} = \frac{(\Delta P_{dil} \cdot V_{dil}) + (\rho_s(1-e_t)V_s) + (\rho_l \cdot e_{dil} \cdot V_{dil}(0.2))}{(\Delta P_d \cdot V_s) + (\rho_s(1-e_t)V_s) + \left(\rho_l \cdot e_t \cdot \left(\frac{V_s + V_{slip}}{100} \right) \cdot 0.2 \right)}$$

where ΔP_{dil} = pressure drop of water flowing at V_{dil} ,

W_{dil} = work done in lifting the solids in dilute phase,

W_{den} = work done in lifting the solids in dense phase,

ρ_s = density of the solids,

$V_{dil liq}$ = dilute phase liquid velocity,

V_{slip} = absolute velocity of liquid past solids in dense phase.

The components of both denominator and numerator in the ratio of work to move solids in the two phases are work against friction (liquid or solids), the work to lift the solids, and the work to lift 20 percent of the water. All pressures are expressed in consistent units of water head. The work units are rather unconventional but this is unimportant in a ratio.

The results of the ratio of work for the two phases are plotted in fig. 47.3 for two values of voidage in dilute phase flow.

7.7. Attrition.

It was naturally difficult to form any firm impressions of the amount of attrition that had taken place with the silver sand but the ballotini showed the same absence of damaged particles (fig. 36) which was noted with the ion-exchange resin beads in Part I. No discernible wear had occurred at the pipe walls even at the right angled bends where the original scores of drilling were still visible even with the brass couplings.

Comparison of work to lift solids in 25% v/v (W_{dil}) with work to lift solids through the same pipe in dense phase flow (W_{den}) at varying solids velocities.

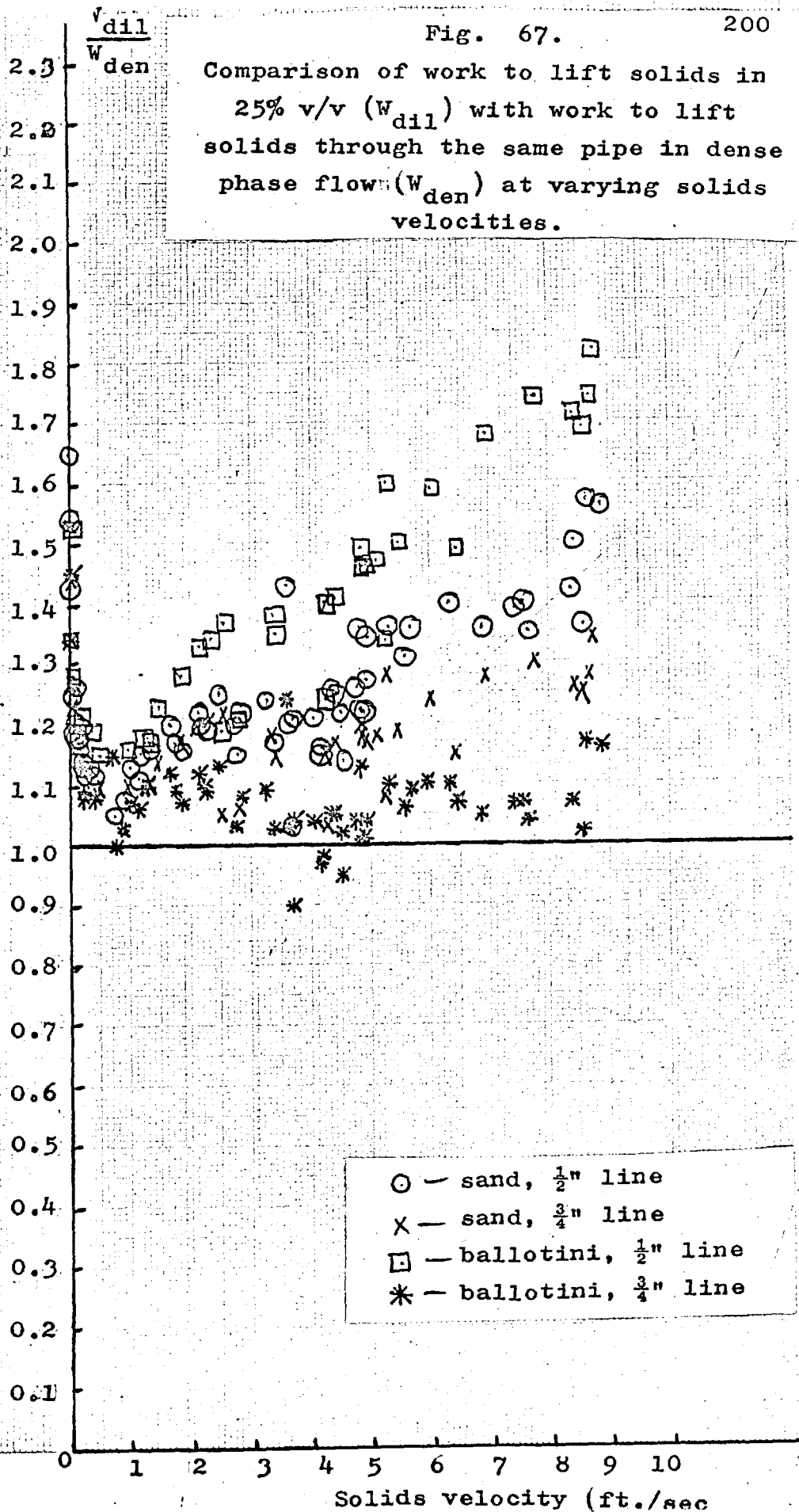
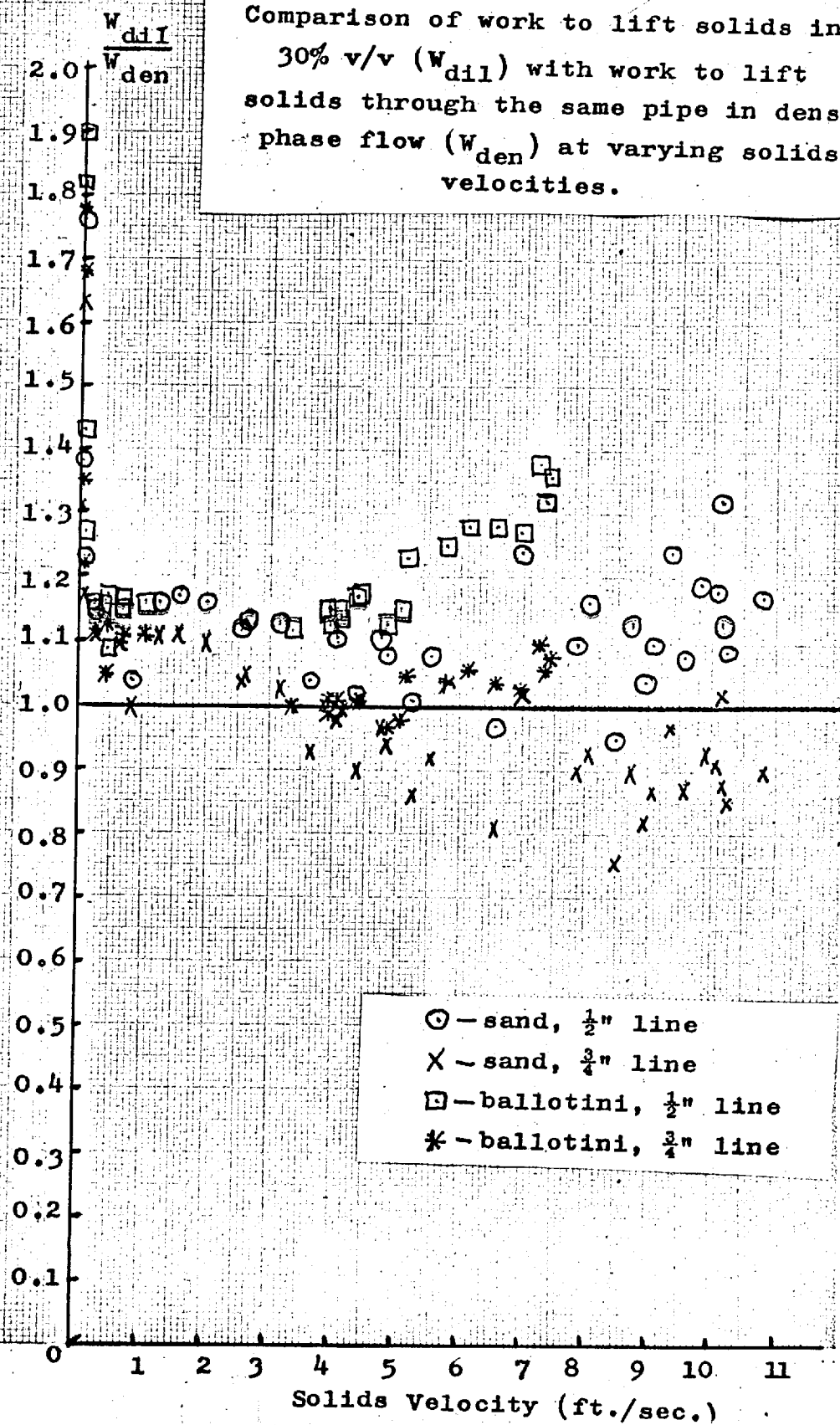


Fig. 68.

Comparison of work to lift solids in 30% v/v (W_{dil}) with work to lift solids through the same pipe in dense phase flow (W_{den}) at varying solids velocities.



8. DISCUSSION AND CONCLUSIONS.

8.1. Discussion.

In their experiments in low velocity dense phase flow, Hancher and Jury (48) and Hershey and Peebles (49) dealt with a complex system. However, it may be helpful to consider the complications which arose in their study in comparison with this investigation.

The American workers studied solids contained within a short length of column and solids pressure varied continuously through the length of the solids bed. In the experiments of this study, the flow was undisturbed over long sections before and after the pressure measurement section and it could confidently be assumed that solids pressure would be constant with the drag forces on the solids in equilibrium with frictional and gravitational forces.

The two American studies used ion-exchange resin - water systems and this introduced a further complication. They considered that the compressible solids particles would be distended at the pipe walls by solids pressure. In that state, liquid pressure would be added to the solids pressure in forcing the

solids against the walls. However, neither sand nor ballotini are appreciably distended by solids pressures of less than the magnitude causing fracture and liquid pressures can be taken as acting in all directions on even those particles closest to the walls. However, it was observed that the dense phase flow of sand in P.V.C. tubing gave very large pressure drops and liquid pressure might well be transmitted to walls of so compressible a material.

The remaining forces acting on the solids particles in dense phase flow, can be treated as uniform through the pressure-tapped test section. Two forces act upward on solids particles. These are the drag force of the slip water and the buoyancy of the water. This latter force has been eliminated by considering the gravitational force to act on the apparent weight of solids in water. The drag force is balanced by the two downward acting forces of gravitational attraction on the apparent weight of the solids and friction.

The studies of Hancher and Jury and Hershey and Peebles assumed that the frictional losses in dense phase flow of resin occurred between the solids and the

pipe walls and were in large measure directed towards determining the coefficient of friction with various externally applied solids pressures. Nonetheless, solids pressure cannot be assumed to exist unless a solids-wall friction force can be demonstrated. But it is not difficult to conceive solids pressure with the high solids packings encountered where the solids were more closely packed than in the free-settled state.

A liquid friction pressure drop seems most unlikely since solids velocity correlates with pressure drop much more linearly than the more accurately measured mean velocity of liquid and solids. Use of liquid velocity would increase the scatter of the poorer correlation using mean velocity. Further, the linear nature of friction pressure losses is quite unlike those of fluid flow.

If the frictional pressure losses are predominantly solids-solids, they might either result from friction between solids particles and the walls or from friction between two solids particles. Frictional losses at the walls seem rather more likely since observation of dense phase flow in transparent tubes suggests little relative motion of solids particles.

Assuming the existence of solids pressure and a solids-wall friction mechanism for frictional pressure drop, the forces acting in an element of pipe containing dense phase flow are:

$$\text{Wall friction force on solids} = \mu P_s \pi D(1-e_t)dh,$$

Applied solids - liquid drag force,

nett of work in lifting solids against

$$\text{gravity} = \Delta P_d \cdot dh \cdot \pi D^2/4,$$

where μ = coefficient of solids-wall friction,

P_s = pressure of solids,

D = tube diameter,

e_t = voidage in the tube,

dh = height of the tube element,

ΔP_d = dynamic pressure drop over unit length.

Equating these two forces,

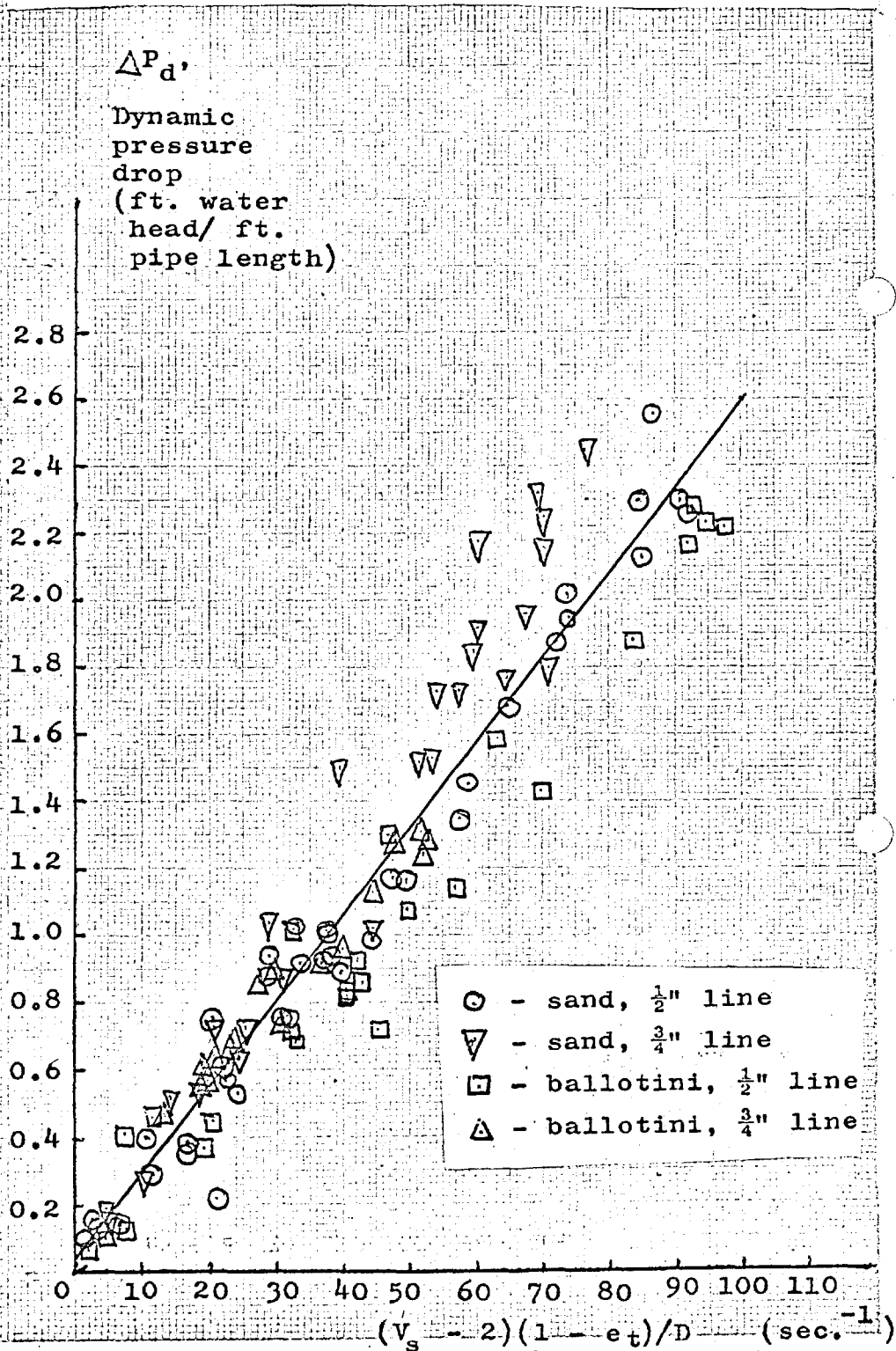
$$\Delta P_d \cdot dh \cdot \pi D^2/4 = \mu P_s \pi D(1-e_t)dh$$

$$\therefore \Delta P_d = 4\mu P_s (1-e_t)/D$$

Plotting dynamic pressure drop against solids velocity multiplied by solids fraction and divided by tube diameter shows that dynamic pressure drop varies inversely with the first power of tube diameter. (fig. 48) This relationship of dynamic pressure drop and diameter could hardly arise from interparticle friction which would occur

Fig. 48.

Dynamic pressure drop plotted against the velocity of solids in excess of 2 feet/sec. multiplied by the solids fraction divided by the tube diameter.



throughout the cross-section of the tube and establishes a wall friction mechanism for frictional pressure drop which also implies the existence of solids pressure. The solids pressure would appear to vary directly with the solids velocity. The effect of solids fraction could not be established decisively since the correlation of dynamic pressure drop with solids velocity divided by diameter was as good as the correlation with solids velocity multiplied by solids fraction and divided by diameter. The correlation coefficient for this plot was 0.959. The correlation coefficient without multiplying by the solids fraction was 0.969 but some extra error is introduced with the additional factor.

Without direct measurement of either the coefficient of friction between the solids and the walls of the tube or the pressure of the solids, it is impossible to separate these two factors. Direct measurement would seem to present considerable difficulties. It would be difficult to reproduce the conditions of dense phase flow in a rotational viscometer and this would seem valuable only to confirm direct measurement of solids pressure. Direct measurement of the force acting on the tube walls could be made but no simple method presents

itself. No experiments to measure either of these factors separately were made partly because of the difficulties of devising suitable experiments and partly because the information seemed unlikely to serve any useful purpose as the coefficient of friction would presumably be constant and the solids pressure dependent on solids velocity.

The co-incidence of the plots of dynamic pressure drop against solids velocity for the two very differently shaped solids particles is noteworthy. Wen (31) in studies of horizontal solids-gas dense phase flow found the same independence from particle shape.

That solids-wall friction does not exist at solids velocities below the sharp transition about 2 feet/sec. is indicated by the close concurrence of the dynamic pressure drop and the plot of pressure drop with water only flowing when these are plotted against mean velocity. In this region, the pressure drop follows the normal pattern of dilute phase flow.

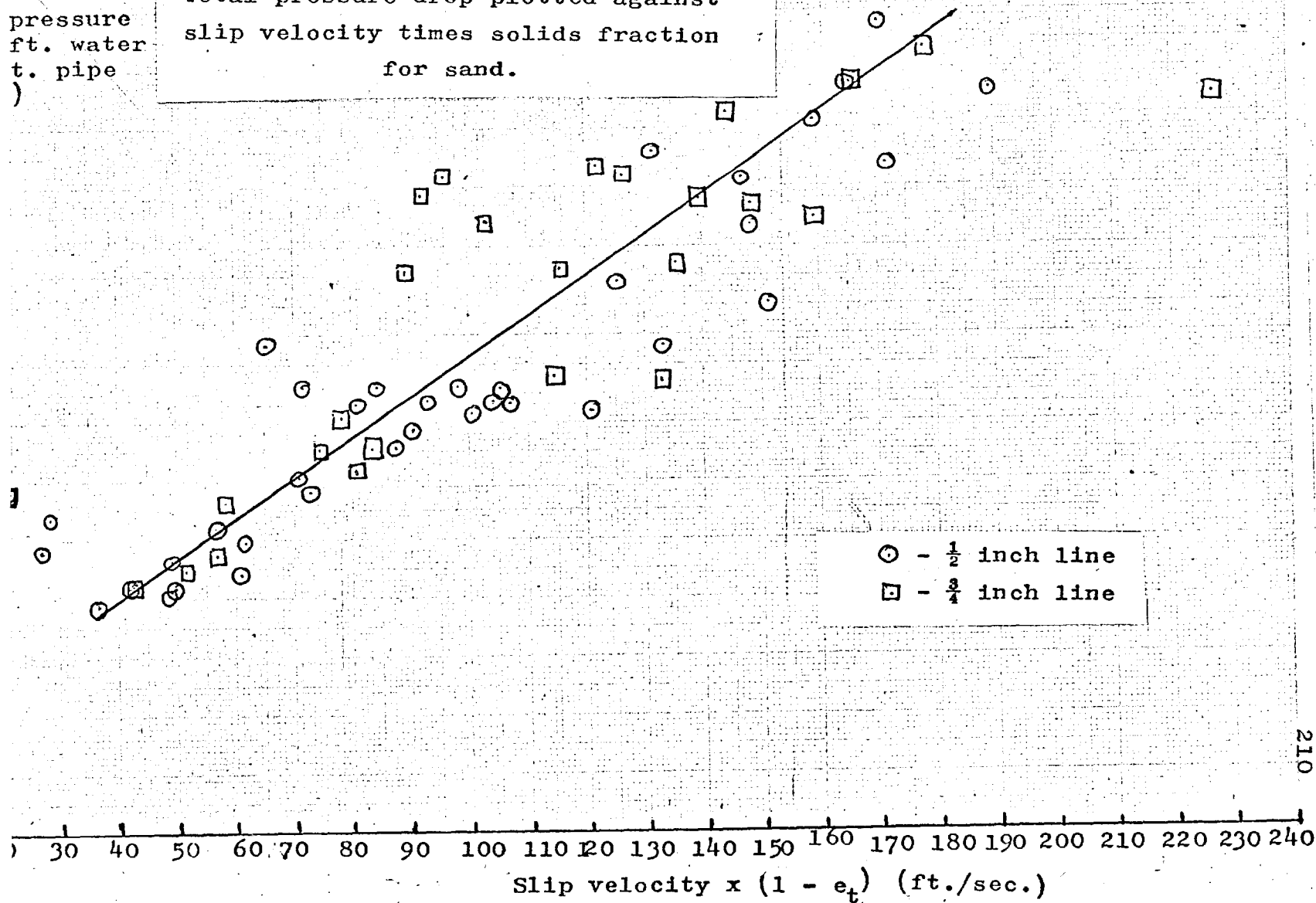
Consider now the drag force acting on the solids as a result of slip water flow. The force exerted on the individual solids particles depends on the drag coefficient, an effective diameter of the particle (which allows for

irregularities of shape), the velocity of the slip water, and the viscosity and density of the water. With any one liquid and solids particle, at constant relative velocity between liquid and solid particle, the drag force on the particle will be the same. Now the drag force on the particle assembly in dense phase flow is measured as the total pressure drop and the number of particles experiencing this force is proportional to the solids fraction. Hence, a plot of slip velocity multiplied by solids fraction ought to correlate with total pressure drop regardless of the solids velocity. (Fig. 65 and 66) Within the wide margins of experimental error associated with slip rate, this appears to be true, though as with the correlation of dynamic pressure drop, the presence or absence of solids fraction does not materially affect the correlation. This explains why the slip ratio, while being a constant fraction of solids velocity in any one tube and with one solids material, is significantly reduced in the larger diameter tube where the solids velocity must be greater than in the smaller tube to produce the same frictional resistance. The two solids materials have already been shown to have slightly different drag coefficients (fig. 49) which is not surprising in view

Fig. 65.

Total pressure drop plotted against
slip velocity times solids fraction
for sand.

pressure
ft. water
t. pipe
)



ΔP_t ,

Total pressure drop
(ft. water head/ft.
pipe length)

Fig. 66.

Total pressure drop plotted against
slip velocity times solids fraction
for ballotini.

○ - $\frac{1}{2}$ inch line
□ - $\frac{3}{4}$ inch line

Slip velocity $\times (1 - e_t)$ (ft./sec.)

of their difference in shape but substantiates the conclusion that particle shape had little effect on wall friction.

The plot of drag coefficient, C_D , and particle Reynolds number (fig. 49) ^{appears to} shows two regimes divided by the transition at ^{a Reynolds number of about 100} (about 2 feet/sec.). The nature of the dense phase flow must change radically at the transition for it to acquire a solids pressure and some change in the drag coefficient would be expected. However, it is rather strange that the change is such as to allow the slip ratio and solids fraction to stay constant when, above the transition, work must be done on the solids to overcome the additional force of wall friction.

The drag coefficient on the individual solids particles varies as the inverse second power of velocity below the transition (that is, the drag force is independent of the velocity) in the same manner as described by Wen (31) for solids-gas dense phase flow. Above the transition, the drag coefficient varies as the inverse first power of the velocity. This is in the normal manner of laminar flow but the coefficient is much smaller than indicated by Stokes Law and the velocity above the normal range of laminar flow. The

pressure drop is not of course characteristic of a packed bed which would give a much higher pressure drop at the same slip velocity. This was observed to occur in the experiments with an expanded section of pipe in the $\frac{1}{2}$ inch line.

The experiments with the expansion and contraction in the $\frac{1}{2}$ inch line are exceedingly informative. The requirement of a slip velocity in the narrow section of the flow line sufficient to maintain fluidized conditions in the widest section is a logical explanation of the "jamming" observed at lower flow rates. The excellent correlation between the minimum slip ratio to avoid jamming and the observed minimum fluidizing velocity is both the evidence for this theory and substantiation of the technique of measuring slip ratio and transmitted solids voidage.

"Jamming" arises when the minimum fluidizing velocity is not maintained and to restore fluidization requires restoration of this velocity but with the flow now passing through the narrow section as flow through a packed bed and not as the slip water in dense phase flow conditions. It has already been noted that dense phase flow gives much reduced liquid-solids drag forces. Hence, the pressure required will be much greater where the

expansion is far removed from the entry to the pipeline since the solids downstream from the expansion and contraction will be fluidized long before those in the expansion but those upstream will be restrained by the settled solids in the expanded section. "Jamming" with high pressure drops has been observed in a small solids-liquid contactor of the type described in Part I but the connecting pipework of narrow bore was only a few inches long and the solids could be moved. Only the expansion causes jamming and a contraction to narrower line would have no effect. On the other hand, adding water to the expansion to clear the contraction and the downstream portion of the line has no effect on jamming as was observed with the expansion in the $\frac{1}{2}$ inch line.

The pressure drops occurring at a right angled bend are of limited value especially in view of the peculiarly low pressure drop associated with sand in the $\frac{3}{4}$ inch line. However, the much superior correlation of this pressure drop as a function of the total rather than the dynamic pressure drop is understandable since the force overcoming the pressure drop at the bend is a drag force of the slip water.

The step transition in the overall pressure

drop seems explicable only in terms of the pressure drop at a change from horizontal to vertically upward flow. With the resistance to flow of the right angled bend, a slight increase in the solids fraction must occur and in the cases where the very high overall pressure drops were noted the solids fraction appears to have reached the maximum packed settled fraction. As a result, the phenomenon of high overall pressure drop at low flow rates is much more evident with the ballotini which travelled in dense phase flow with a solids fraction quite close to the maximum, and is most marked with the $\frac{3}{4}$ inch line which had the highest solids fraction of all.

There must be some error in the density gauge determinations caused by this region of increased solids concentration after flow has changed from horizontal to vertically upward at the entry to the density gauge test section. However, the experiments with the $\frac{1}{2}$ inch line show that the slip velocity and hence the solids fraction were reasonably accurate. Further, the co-incidence of the dynamic pressure drop versus mean velocity plot with the water only plot at velocities below about 3 feet/sec. suggests that the static head of solids was not overestimated.

The slight change in slope of the log-log plot of overall pressure drop at a solids velocity of about 5 feet/sec. supports the reality of a slight step discontinuity observed at this velocity in the pressure drops for vertically rising flow. However, this discontinuity is small and ignoring it does not greatly affect the correlation of pressure drop in a straight section of vertically rising flow. The nature of the discontinuity is not understood.

Comparison of dense phase flow with dilute phase flow at 25 and 30 percent solids indicates a modest saving in pumping power using dense phase flow to convey the same volumetric flow of solids through the same pipe. (Fig. 67 and 68). But the advantage of dense phase flow is less at the higher pipe diameter and the pressure drop in dense phase flow has been shown to decrease with the first power of diameter while that for dilute phase (water friction) decreases slightly more rapidly with increasing diameter. Hence, dense phase flow for vertical conveying would not offer any saving in pumping costs with commercial sizes of pipeline though the difference between the two flow states would be small.

Dense phase flow appears to give negligible particle attrition - evident especially in the Part I performance of the extremely friable ion-exchange resin - and insignificant pipe erosion. This aspect of dense phase flow would be worth further study since pipe erosion can be very significant with dilute phase flow.

The mechanism of pressure drop by friction of solids particles sliding along the tube walls is a unique feature of dense phase flow and it would be interesting to see whether this behaves entirely as more conventional forms of friction. If this were so, use of teflon lined tubes or some other low friction material would be expected to produce a reduction in dynamic pressure drop.

Further work might also be done on the behaviour of the more complex horizontal dense phase flow.

Since larger pipe diameters would be impracticable in an experimental apparatus unless the solids could be recycled, measurement of the variation of solids fraction across the height of a pipe carrying horizontal dense phase flow would be difficult. The effect of varying particle size would be investigated.

8.2. Conclusions.

Lifting of solids by dense phase flow has been shown to require about the same amount of work as dilute phase conveying if the same volume of solids is to be conveyed through the same pipe in unit time. The small saving of work with the narrow bore pipes used seems unlikely to arise with large pipes. However, in any process where it is desirable to transfer solids with the minimum of conveying liquid, dense phase flow would be the obvious choice.

Another case in which dense phase flow would offer advantages would be where it was impracticable to recover the potential energy of the liquid conveying the solids. Failure to recover this potential energy is not of great consequence with dense phase flow but with dilute phase flow about half the work will be expended in lifting the conveying liquid. This relative unimportance of recovering liquid potential energy is typical of the simplicity of dense phase flow.

If pipe erosion were shown to be insignificant over longer periods than have been possible in this study, dense phase flow could be very attractive for solids conveying operations where severe pipe erosion

is caused in dilute phase conveying or where it was important to avoid contamination of the flowing solids with fragments of pipe material.

This investigation has shown that the commercially accepted 35 percent solids fraction by volume limit on pipeline conveying need not be observed if the pipeline is designed to be free from marked variation in diameter. Because of the importance of avoiding marked expansions in the flow channel, direct pumping of dense phase flow would probably be difficult and might require a specially designed pump. However, over short distances, the procedure adopted in these experiments of pumping conveying liquid into the feed vessel could be used. Two feed vessels might be filled and pumped free alternately.

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Appendix 1. Characteristics of the solids.

The weight fractions estimated by the method discussed in section 2.1.2, are listed in table 1.

Incipient fluidization velocities and the voidages of fluidized beds were measured using a 2 inch glass column with a cloth distributor. Flow rates were measured by a rotameter and the pressure upstream from the cloth distributor was measured with an ethylene dibromide - water manometer. A series of pressure measurements were carried out at varying flow rates with water only in the column. A known weight of solids was then introduced to the column and the lowest flow rate at which the pressure drop exceeded the water only pressure drop by the apparent static head of solids was noted. This is the incipient fluidization velocity.

With ion-exchange resin, the point of incipient fluidization was indicated by a fairly definite point of inflexion on the plot of pressure drop against flow rate. With the heavier solids, it was marked by a sharp discontinuity in this plot. The flow rates of incipient fluidization are listed in table 2.

Particle Reynolds numbers and drag coefficients were calculated and used with voidages to compare the results with published data on fluidization (33). It was found that the results for each solids material corresponded to a definite particle size in the published data. These particle sizes which can be regarded as a characteristic size for the fluidization properties of each solids material are listed in table 2.

Voidage
of solids
bed

Fig. 64 .

Variation of voidage in an evenly fluidized
solids bed with absolute liquid velocity.

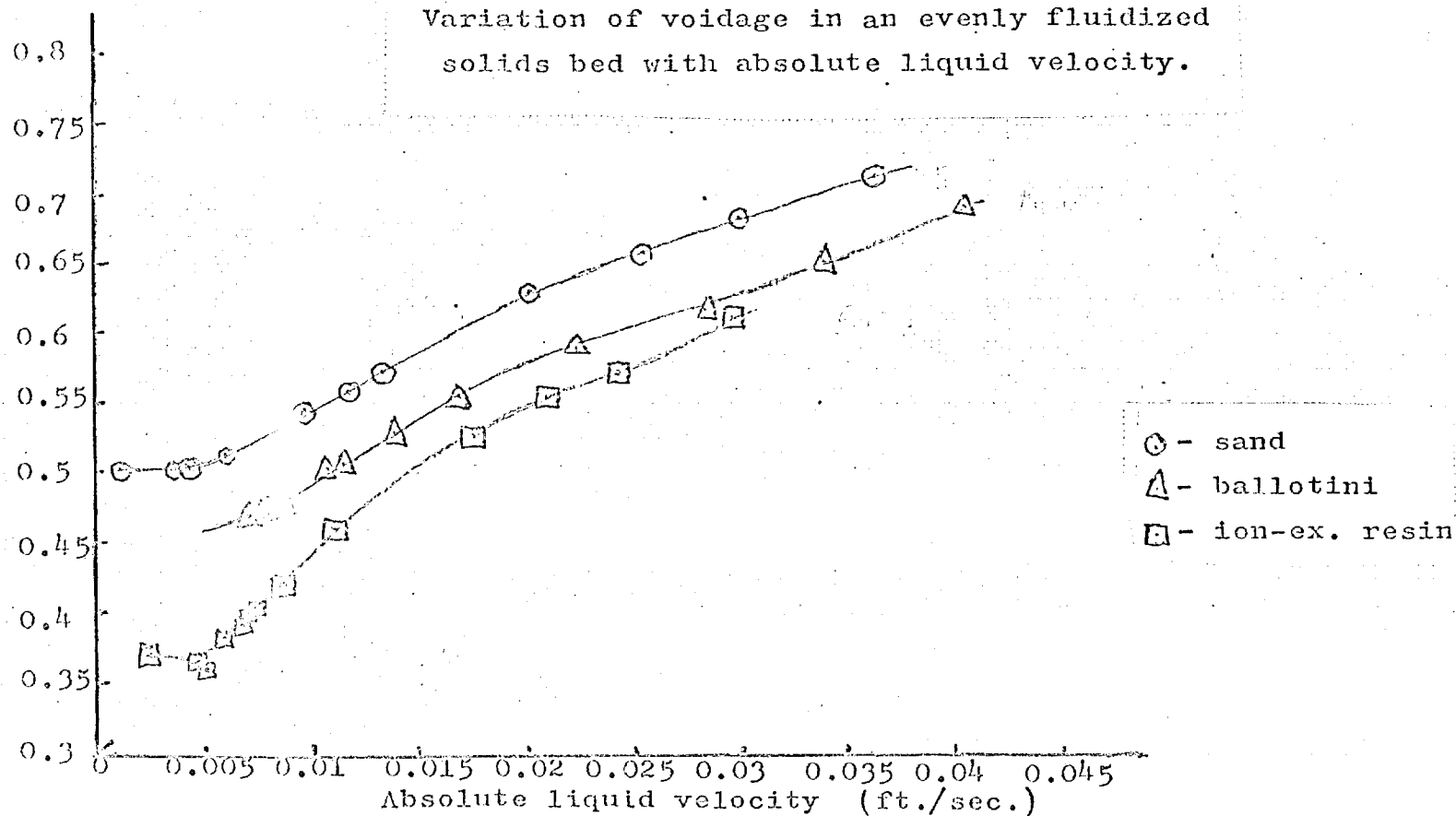


Table 1. Photographically estimated size distribution of the solids materials.

B.S.S. mesh size	Estimated weight % of sample passing mesh size		
	Sand	Ballotini	Resin
14	100	100	100
16	100	100	90.1
18	100	100	79.2
22	100	100	60.6
25	94.9	87.0	35.9
30	90.2	81.6	19.0
36	76.1	65.7	2.1
44	42.3	17.9	0
52	17.2	3.3	0
60	9.7	1.0	0
72	1.6	0.4	0
85	0.2	0	0
100	0	0	0

Table 2. Physical properties of solids materials.

	Sand	Ballotini	Resin
Density (gm./ml.)	2.646	2.498	1.295
Tap-settled voidage	0.383	0.385	0.325
Free-settled voidage	0.489	0.439	0.370
Incipient fluidization velocity (ft./sec.)	0.0087	0.0080	0.0090
Characteristic particle size of fluidized properties (in.)	0.0122	0.0146	0.036

Appendix 2 - Variance Analysis of Factorial Experiments.

The investigation must be performed with equally spaced levels of the independent variables. The use of equal spacing allows the use of orthogonal polynomials to make the estimates of variance (due to an effect) and co-variance (due to an interaction) orthogonal - i.e. mutually independent. Without orthogonality, the variances produced by analysis are the sum of variances and co-variances from several effects. The uses of orthogonal polynomials is widely described (56) and the values listed (57). The layout used in the analyses is due to Dr. P. Eisenklam of Imperial College.

Method of Analysis.

A typical $3 \times 2 \times 2$ (3 levels of one variable and two each of two other variables) analysis is set out in table 3. The statistics (experimental results) appear in the second column from the left, S. The results may be manipulated by any uniform processes of addition, subtraction, multiplication, or division before analysis and are usually arranged to make the moduli as small as possible without involving a decimal point. The transformed statistics appear in column 3, S'. The extreme left hand column lists the operators for the subsequent

Table 3 - Example of full variance analysis.

Test qrt	S	S'	S' ²	$\bar{Q}$	$\bar{R}$	Total effect	Total square effect	d	Mean square effect	F	Signi- ficance	Mean effect
000	19.5	- 55	3025	- 41	- 79	+ 22	484	24	40:4	-	-	-
100	23.8	- 12	144	- 38	+101	+642	12164	8	51520:6	128.8	0.1%	+80.3
200	27.6	+ 26	676	- 1	+299	+106	11236	24	468:2	-	-	-
010	14.2	-108	11664	+102	+343	+106	11236	12	936:4	-	-	-
110	21.0	- 40	1600	+ 81	+ 77	+234	54756	8	6844:6	17.1	1%	+29.3
210	36.0	+110	12100	+218	+ 29	+130	16900	24	704:2	-	-	-
001	18.7	- 63	3969	+123	+ 3	+180	32400	12	2700:0	6.8	5%	+15.0
101	25.2	+ 2	4	+220	+103	+ 44	1936	8	242:0	-	-	-
201	31.0	+ 60	3600	- 5	+137	- 48	2304	24	96:0	-	-	-
011	18.0	- 70	4900	+ 82	+ 97	+100	10000	12	833:4	-	-	-
111	27.2	+ 22	484	- 7	+ 87	- 40	1600	8	200:0	-	-	-
211	40.0	+150	22500	+ 36	+ 43	- 44	1936	24	80:8	-	-	-
Sum		+ 22	64666	+770	+1240	+1432			64666:0			
Check sum		+770		+1240	+1432							

manipulations and the data are arranged to correspond to these operators. Thus, the statistic for $Q_3R_1T_2$ is entered opposite operators 2 0 1. Taking first the extreme left hand operator, the data are multiplied by the operating coefficient for a three level factor (from tables of orthogonal polynomials) and the sums of groups of three statistics (all at a constant level of R and T) are entered in the column $\bar{Q}$. Column $\bar{Q}$ now lists multiples of the average values of various effects of Q at fixed levels of R and T. Each group of three statistics is operated on three times to assess in turn the average value of S' , linear changes of S' with Q, and curvilinear changes of S' with Q.

In the column headed $\bar{R}$, the data of $\bar{Q}$ is treated in the same way though by operating coefficients for a factor at two levels and treating the values of $\bar{Q}$ twice in pairs. It can be seen that the data in column $\bar{Q}$ alternately represents data at R_1 ($r = 0$) and R_2 ($r = 1$) and so column $\bar{R}$ gives the effect of R at constant Q and T. Finally, using the figures of $\bar{R}$, the total effect column repeats the process used in forming $\bar{R}$. +22 can be seen to be the sum of all the figures in column S' ; R_1 is the difference between the sum of all the statistics

at r_0 and those at r_1 . The effect represented by each figure can be read from operators column. For example, 2 0 1 represents $Q_2'T_1'$, the curvilinear interaction of Q with T.

These total effects are then squared for significance testing and divided by a divisor which is $\frac{grt}{11}$ (sum of squares of the operating coefficients contributing to that total effect). For example, the operating coefficients making up $Q_2'T_1'$ are +1, -2, +1, to give $\bar{Q}$; +1, +1, to give $\bar{R}$; and -1, +1, to give $\bar{T}$. Then the divisor for $Q_2'T_1'$ is $\{(1)^2 + (-2)^2 + (1)^2\} \{(1)^2 + (1)^2\} \{(-1)^2 + (1)^2\} = 24$. The mean square is the total effect squared divided by the divisor. Its departure from zero may be real or due to random errors and to estimate the random error all the small and obviously random squares are pooled to give a residual, $\frac{4}{2}$. (The results may also be compared with an estimated experimental error (squared) and this is a useful check against freak results.) In this case, 9 terms are insignificant and are pooled to give a residual value of 400 (the average value of the mean squares in the residual) and one degree of freedom for every term pooled. Dividing

the remaining individual mean squares by 400 gives the values of F to be compared with the standard values of F at $\nu_1 = 1$, $\nu_2 = 9$ for significance. Any terms which are not significant to the required level are pooled in a new residual.

The check totals are obtained by doing simultaneously all the operations on a column which are carried out individually to produce the next column. The sum of the mean squares must always equal the sum of the squares of the statistics in column S'^2 . (Note. The ':' symbol used in the mean square effect signifies duodecimal notation after the point.) The mean effects column can be used to build a complete regression equation for the significant effects of Q , R , and T on S , and details of the procedure are given by Eisenklam. (58)

Shortened versions of all the analyses of results are given in tables 4 - 10.

Analysis of Variance.

Statistic - $V_{\max}$ for silver sand.

Test qrt	S	S'	$\bar{Q}$	$\bar{R}$	Total effect	Total square effect	d	Mean square effect	Symbol
000	3.3	- 37	+ 55	+107	+272	73984	12	6165.33	$\bar{N}$
100	8.9	+ 19	+ 52	+165	+467	218089	8	27261.13	Q_1
200	14.3	+ 73	+ 85	+216	+ 11	121	24	5.06	Q_2
010	3.7	- 33	+ 80	+251	- 8	64	12	5.4	R_1
110	8.2	+ 12	+110	+ 14	- 9	81	8	10.16	$Q_1 R_1$
210	14.3	+ 73	+106	- 3	+ 31	961	24	40.16	$Q_2 R_1$
001	3.3	- 37	+128	- 3	+ 58	3364	12	280.33	T_1
101	10.1	+ 31	+123	- 5	+ 35	1225	8	153.13	$Q_1 T_1$
201	16.1	+ 91	- 2	- 4	- 17	289	24	12.06	$Q_2 T_1$
011	3.6	- 34	+ 16	- 5	- 2	4	12	0.4	$R_1 T_1$
111	9.5	+ 25	- 8	+ 18	- 1	1	8	0.16	$Q_1 R_1 T_1$
211	15.9	+ 89	+ 5	+ 13	- 5	25	24	1.06	$Q_2 R_1 T_1$
Sum		+272	+750	+764	+832			33934	
Check sum		+750	+764	+832					

Residual mean squares is 4.863 with six degrees of freedom.
 i.e. Mean squares of greater than 29.13 are significant to 5%.
 Mean squares greater than 66.82 are significant to 1% level.
 Mean squares greater than 172.69 are significant to 0.1%.

Estimated error in determining $V_{\max}$ is equivalent to 10 units of S' arising entirely from errors in setting Q. i.e. Error estimated residual mean square is 100 with 11 degrees of freedom.

Bartlett tests indicate less than 20% probability that the departure of the data from normal distribution between the levels of Q arose from random variation. The departure of data from normal distribution between the levels of R had a 99.5% probability of origin from random variation.

Analysis of Variance.

Statistic - H_{crit} for silver sand.

Test qrt	S	S'	$\bar{Q}$	$\bar{R}$	Total effect	Total square effect	d	Mean square effect	Symbol
000	19.5	- 55	- 41	- 79	+ 22	484	24	40:4	M
100	23.8	- 12	- 38	+101	+642	412164	8	51520:6	Q_1
200	27.6	+ 26	- 1	+299	+106	11236	24	468:2	Q_2
010	14.2	-108	+102	+343	+106	11236	12	936:4	R_1
110	21.0	- 40	+ 81	+ 77	+234	54756	8	6844:6	Q_1R_1
210	36.0	+110	+218	+ 29	+130	16900	24	704:2	Q_2R_1
001	18.7	- 63	+123	+ 3	+180	32400	12	2700:0	T_1
101	25.2	+ 2	+220	+103	+ 44	1936	8	242:0	Q_1T_1
201	31.0	+ 60	- 5	+137	- 48	2304	24	96:0	Q_2T_1
011	18.0	- 70	+ 82	+ 97	+100	10000	12	833:4	R_1T_1
111	27.2	+ 22	- 7	+ 87	- 40	1600	8	200:0	$Q_1R_1T_1$
211	40.0	+150	+ 36	+ 43	- 44	1936	24	80:8	$Q_2R_1T_1$
Sum		+ 22	+770	1240	1432			64666	
Check sum		+770	1240	1432					

Residual mean square is 400 with 8 degrees of freedom.

Mean squares greater than 10168 are significant to 0.1% level.

Mean squares greater than 4504 are significant to 1% level.

Mean squares greater than 2128 are significant to 5% level.

Estimated error in determining H_{crit} is equivalent to 20 units of S' from setting errors in Q and 5 units of S' in determining H_{crit} from the graphs. i.e. Error estimated residual mean square is 625 with 11 degrees of freedom. Bartlett test indicates an approximately 50% probability that departure of the data from the normal distribution between the levels of Q arose from random variation.

Analysis of Variance.

Statistic - operating gradient, m, for silver sand.

Test qrt	S	S'	$\bar{Q}$	$\bar{R}$	Total effect	Total square effect	d	Mean square effect	Symbol
000	0.29	-21	- 1	-15	- 49	2401	12	200.1*	
100	0.51	+ 1	-14	-34	+141	19881	8	2485.16	Q_1
200	0.69	+19	+ 9	+67	- 7	49	24	2.06	Q_2
010	0.31	-19	-43	+74	- 65	4225	12	352.1*	R_1
110	0.47	- 3	+40	- 9	+ 15	225	8	28.16	Q_1R_1
210	0.58	+ 8	+27	+ 2	+ 25	625	24	26.06	Q_2R_1
001	0.32	-18	+36	+13	- 19	361	12	30.1	T_1
101	0.57	+ 7	+36	-52	+ 7	49	8	6.16	Q_1T_1
201	0.70	+20	- 4	-13	+ 11	121	24	5.06	Q_2T_1
011	0.20	-30	- 5	- 2	- 39	1521	12	126.9*	R_1T_1
111	0.31	-19	-12	- 1	+ 11	121	8	15.16	$Q_1R_1T_1$
211	0.56	+ 6	+14	+26	+ 27	729	24	30.46	$Q_2R_1T_1$
Sum		-49	+85	+30	+ 28			3307	
Check Sum		+85	+30	+28					

Residual mean square is 17.87 with 7 degrees of freedom.
Mean squares greater than 522.7 are significant to 0.1% level.
Mean squares greater than 218.9 are significant to 1% level.
Mean squares greater than 99.9 are significant to 5% level.
No error estimate of the residual mean squares is available.
Bartlett test indicates a slightly less than 70% probability
that departure of the data from normal distribution between
the levels of Q arose from random variation.

Analysis of Variance.

Statistic - $V_{\max}$ for ion-exchange resin.

Test qrt	S	S'	$\bar{Q}$	$\bar{R}$	Total effect	Total square effect	d	Mean square effect	Symbol
000	22.7	-373	+122	+201	+661	436921	12	36410:1	Q_1
100	66.0	+ 60	+179	+360	3192	10188864	8	1273608	
200	103.5	+435	+178	1599	+ 10	100	24	4:2	
010	27.3	-327	+182	1593	+ 61	3721	12	310:1	R_1
110	64.2	+ 42	+808	- 5	- 22	434	8	60:6	Q_1R_1
210	106.4	+464	+791	+ 15	+ 83	7744	24	322:8	Q_2R_1
001	26.3	-337	+799	+ 57	+ 59	3481	12	290:1	T_1
101	65.3	+ 53	+794	+ 4	- 6	36	8	4:6	Q_1T_1
201	106.2	+462	- 58	- 17	+ 20	400	24	16:8	Q_2T_1
011	26.3	-337	+553	- 5	- 53	2809	12	234:1	R_1T_1
111	66.2	+ 62	+ 19	+111	+ 12	144	8	18:0	$Q_1R_1T_1$
211	105.7	+457	- 4	- 23	-134	17956	24	748:2	$Q_2R_1T_1$
Sum		+661	3863	3990	3888			1312027	
Check Sum		3863	3990	3888					

Residual mean squares is 236.3 with eight degrees of freedom.
Mean squares greater than 6006.8 are significant to 0.1% level.
Mean squares greater than 2660.8 are significant to 1% level.
Mean squares greater than 1257.1 are significant to 5% level.
Estimated error in determining $V_{\max}$ is equivalent to 12 units of S' arising mainly from errors in setting Q . i.e.
Error estimated residual mean square is 144 with 11 degrees of freedom.

Bartlett test indicates a 70% probability that departure of the data from normal distribution between the levels of Q arose from random variation.

Analysis of Variance.

Statistic - H_{crit} for ion-exchange resin.

Test qrt	S	S'	$\bar{Q}$	$\bar{R}$	Total effect	Total square effect	d	Mean square effect	Symbol
000	113	-37	+ 7	+ 9	+ 14	289	12	24.1	
100	156	+ 6	+ 2	+ 8	+311	96721	8	12090.16	Q_1
200	188	+38	+12	+153	- 13	169	24	7.06	Q_2
010	112	-38	- 4	+158	- 21	441	12	36.9	R_1
110	150	0	+75	- 9	+ 1	1	8	0.16	$Q_1 R_1$
210	190	+40	+78	- 4	+ 9	81	24	3.46	$Q_2 R_1$
001	114	-36	+80	- 5	- 1	1	12	0.1	T_1
101	154	+ 4	+78	- 16	+ 5	25	8	3.16	$Q_1 T_1$
201	194	+44	-11	+ 3	+ 5	25	24	1.06	$Q_2 T_1$
011	109	-41	+ 2	- 2	- 11	121	12	10.1	$R_1 T_1$
111	150	0	0	+ 13	- 5	25	8	3.16	$Q_1 R_1 T_1$
211	187	+37	- 4	- 4	- 17	289	24	12.06	$Q_2 R_1 T_1$
Sum		+17	+315	+304	+280			12191	
Check Sum		+315	+304	+280					

Residual mean square is 4.448 with 8 degrees of freedom.

Mean squares greater than 113.1 are significant to 0.1% level

Mean squares greater than 50.1 are significant to 1% level.

Mean squares greater than 23.7 are significant to 5% level.

Estimated error in determining H_{crit} is equivalent to 2 units of S' arising almost entirely from setting errors in Q. i.e. Error estimated residual mean square is 4 with 11 degrees of freedom.

Bartlett test indicates a probability of greater than 90% that departure of data from a normal distribution between the levels of Q arose from random variation.

Analysis of Variance.

Statistic - operating gradient, m, for resin.

Test qrt	S	S'	$\bar{Q}$	$\bar{R}$	Total effect	Total square effect	d	Mean square effect	Symbol
000	0.23	-17	+ 7	+14	+ 38	1444	12	120.33	
100	0.45	+ 5	+ 7	+24	+137	18769	8	2346.16	Q_1
200	0.59	+19	+14	+71	- 31	961	24	40.06	Q_2
010	0.24	-16	+10	+66	- 4	16	12	1.4	R_1
110	0.44	+ 4	+36	-13	+ 3	9	8	1.16	$Q_1 R_1$
210	0.59	+19	+35	-18	- 1	1	24	0.06	$Q_2 R_1$
001	0.28	-12	+31	0	+ 10	100	12	8.4	T_1
101	0.47	+ 7	+35	- 4	- 5	25	8	3.16	$Q_1 T_1$
201	0.59	+19	- 8	- 1	- 5	25	24	1.06	$Q_2 T_1$
011	0.24	-16	- 5	+ 4	- 4	16	12	1.4	$R_1 T_1$
111	0.47	+ 7	- 7	+ 3	+ 5	25	8	3.16	$Q_1 R_1 T_1$
211	0.59	+19	-11	- 4	- 7	49	24	2.06	$Q_2 R_1 T_1$
Sum		+38	+144	+142	+136			2528	
Check Sum		+144	+142	+136					

Residual mean square is 2.39 with 8 degrees of freedom.
Mean squares greater than 60.8 are significant to 0.1% level.
Mean squares greater than 26.9 are significant to 1% level.
Mean squares greater than 12.7 are significant to 5% level.
No error estimate of the residual mean squares is available.
Bartlett test indicates a greater than 90% probability that
departure of the data from normal distribution between the
levels of Q arose from random variation.

Analysis of Variance.

Statistic - Retainer utilization for resin.

Test qrt	S	S'	Q	R	Total effect	Total square effect	d	Mean square effect	Symbol
000	.90	+4	+ 6	+ 3	0	0	12	0	
100	.90	+4	- 3	- 3	- 9	81	8	10:16	Q_1
200	.84	-2	+ 7	- 7	-15	225	24	9:46	Q_2
010	.85	-1	-10	- 2	-26	676	12	56:4	R_1
110	.86	0	- 6	- 9	+ 3	9	8	1:16	Q_1R_1
210	.84	-2	- 1	- 6	+ 1	1	24	0:06	Q_2R_1
001	.88	+2	0	- 9	- 6	36	12	3	T_1
101	.89	+3	- 2	-17	+ 5	25	8	3:16	Q_1T_1
201	.88	+2	- 6	+ 5	+ 3	9	24	0:46	Q_2T_1
011	.83	-3	- 3	- 2	- 8	64	12	5:4	R_1T_1
111	.84	-2	- 2	+ 3	- 7	49	8	4:16	$Q_1R_1T_1$
211	.81	-5	- 4	- 2	- 5	25	24	1:06	$Q_2R_1T_1$
Sum		0	-24	-46	-64			96	
Check Sum		-24	-46	-64					

Residual mean square is 3.606 with eleven degrees of freedom.
Mean squares greater than 71 are significant to the 0.1% level.
Mean squares greater than 34.8 are significant to 1% level.
Mean squares greater than 17.5 are significant to 5% level.
Bartlett test indicates a greater than 99% probability that
the departure of the data from the normal distribution
between the levels of Q arose from random variation.

Appendix 3.

Details of calibration of density gauge.

Fig. 69 shows the straight line nature of the plot of $\log_e (I_{sw}/I_{ov})$ against "depth" of solids for both sand and ballotini. Lines of best fit were calculated for both materials using only the data at solids "depths" of greater than 9.5 cm. since lines of best fit calculated from the full range of data would have been disproportionately biased by the scattered points at lower solids "depths". The data in the higher range represented the full variation in ~~solids~~ fraction encountered under flowing conditions. As would be expected from the theoretical linearity of the correlation, the lower points lie very close to the line of best fit calculated from the higher points.

The fitted equations are:

$$- \log_e (I_{sw}/I_{ov}) = 0.07778 x_s + 1.395 \text{ for silver sand,}$$

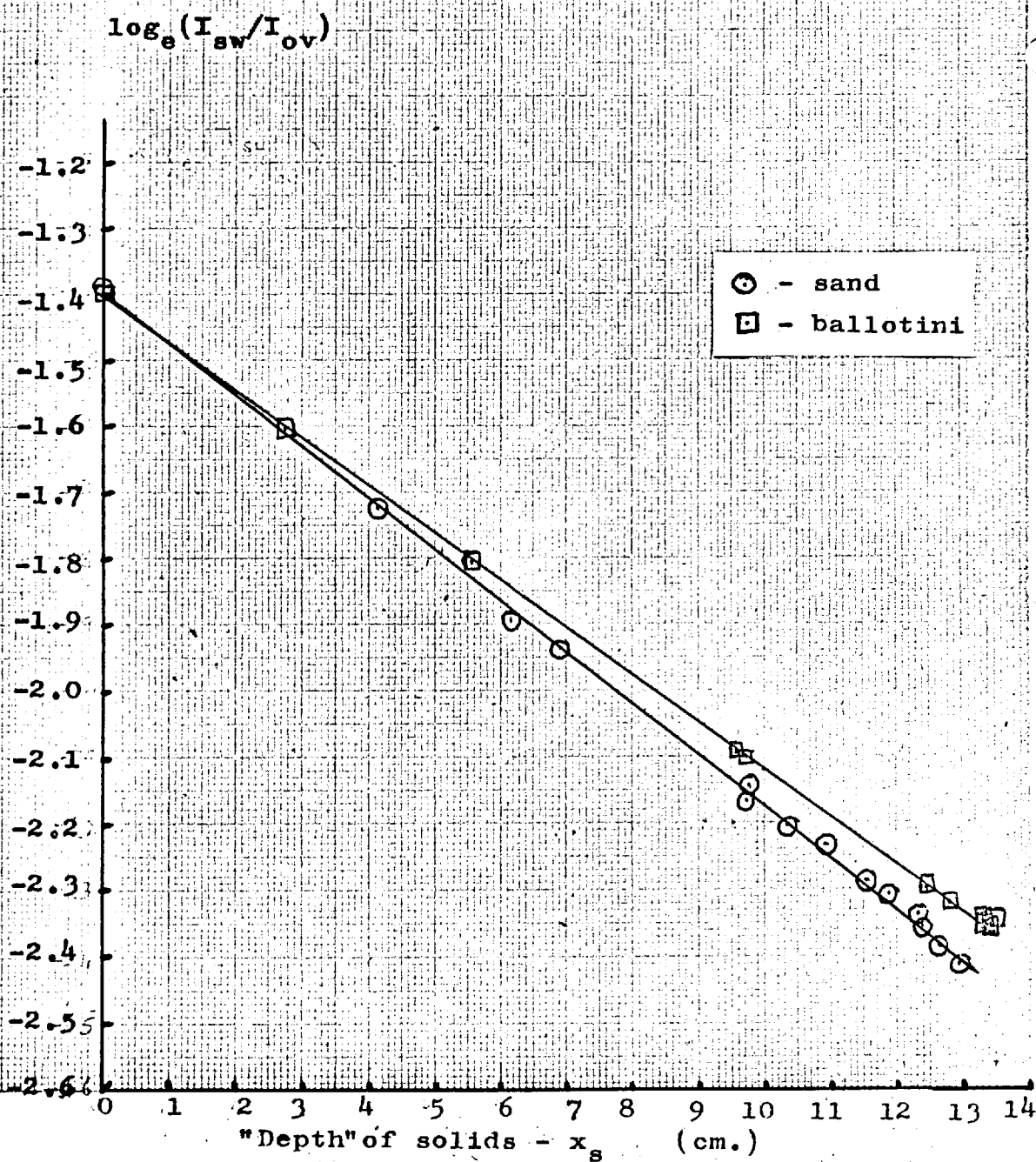
$$- \log_e (I_{sw}/I_{ov}) = 0.71465 x_s + 1.405 \text{ for ballotini.}$$

The theoretical equation is:

$$- \log_e (I_{sw}/I_{ov}) = \mu_s / \rho_s x_s + \mu_w / \rho_w x_w$$

Substituting in the derived equations gives mass

Fig. 69.
Calibration plot of the attenuation
of γ -rays by varying "depth" of
solids.



absorption coefficients of 0.0532, 0.0541, and 0.0633 for silver sand, ballotini, and water, respectively. (The last is the average of the two values of mass absorption coefficient and is in agreement with direct measurement of the coefficient with the calibration tube filled with water.) The densities of silver sand and ballotini are 2.646 and 2.498 gm./cm.³, respectively. These values of the absorption coefficients are in general agreement with the values given for elements by Berry. (55)

In applying the calibration to measurement of voidage of flowing solids, the fitted equations are used directly.

The symbols have the same meanings as used in section 6.1.

Appendix 4.

Sample calculation on dense phase flow.

Levels in metering cylinder: Start liquid	= 63.1 cm.
Start solids	= 5.4 cm.
End liquid	=186.9 cm.
End solids	=114.35cm.
γ-count in 100 sec. with water in test section	= 52407, 51880, 51937, 51835.
γ-count in 100 sec. with dense phase flow	= 21981, 21758.
Start run time	= 2065.00 sec.
End run time	= 2113.39 sec.
Pressure drop over straight section, ΔP	=15.9 cm.Hg/H ₂ O
Pressure recorded in pressure-storage vessel, ΔP	= 24 p.s.i.g.
Pressure recorded at lower tapping point, P ₃	= 10.5 p.s.i.g.
Paralysis time (all runs) on counter, t _p	= 200 μ sec.

Correction of counts for paralysis

$$= \frac{\text{Indicated counts/sec.}}{(1 - (t_p))(\text{Indicated counts/sec.})}$$

Attenuation of water in test section, A _w	= 0.24595
Mass absorption coefficient for water, μ _w	= 0.0633
Mass absorption coefficient for ballotini, μ _b	= 0.071465
Free-settled solids fraction for ballotini, f _b	= 0.5605
Diameter of the ½" pipe	= 0.041238 ft.

No. of right angled bends in the pipeline, B = 4

Density of ballotini, ρ_b = 2.4978

Density of water at 20°C, ρ_w = 0.998

Mean velocity of liquid and solids in pipe

$$= \frac{\text{Level change in metering cyl.} \times \text{vol./cm. cyl.}}{\text{Duration of run} \times \text{X-sectional area of flow}}$$

(for $\frac{1}{2}$ " line)

$$= \frac{\text{Level change in metering cylinder} \times 2.2585}{\text{Duration of run}} \text{ ft./sec.}$$

Static pressure correction factor for straight section

of DP meas., S_c

$$= \frac{\text{length of section} \times (\rho_b - \rho_w) \times 2.54}{(\rho_{\text{Hg}} - \rho_w)} = 13.947 \text{ cm. Hg/H}_2\text{O}$$

Change in liquid level in metering cylinder, L_l = 123.8 cm.

Change in solids level in metering cylinder, L_s = 108.95 cm.

Duration of run, t = 48.39 sec.

Average corrected count rate for water, I_w = 580.54 c.p.s.

Av. corrected count rate for dense phase flow, I_{sw} = 228.70 c.p.s.

$$I_{sw}/I_w = 0.39394$$

$$\text{Depth of solids} = -(\log_e(I_{sw}/I_w))/\mu_b = 13.03 \text{ cm.}$$

$$\text{Path length in test section} = -(\log_e(A_w))/\mu_w = 22.16 \text{ cm.}$$

$$\text{Flowing solids concentration, } (1 - e_t) = 13.03/22.16$$

$$= 0.5880$$

$$\text{Delivered solids concentration, } (1 - e_d) = \frac{L_s \times f_b}{L_1}$$

$$= 0.4933$$

$$\text{Mean flow rate} = \frac{L_1 \times 2.2585}{t} = 5.778 \text{ ft./sec.}$$

$$\text{Solids flow rate} = \frac{L_s \times f_b \times 2.2585}{t \times (1 - e_t)} = 4.847 \text{ ft./sec.}$$

Total pressure drop for vertically rising flow

$$= DP \times 0.99254 \times 0.10759$$

(in ft. water head/ft. pipe length - 0.99254 is a scale correction)

$$\text{(i.e. Total pressure drop, } \Delta P_t) = 1.70$$

Dynamic pressure drop for vertically rising flow, ΔP_d ,

$$= ((DP \times 0.99254) - (S_c)(1 - e_t)) \times 0.10759 = 0.815 \text{ ft./ft.}$$

$$P3_t = P3 - (4.73 \times 0.4335), = 8.4 \text{ p.s.i.g.}$$

(Gauge was 4.73 ft. above P_3 tapping)

$$\text{Total overall pressure drop, } ODP_t = ODP - (4.0 \times 0.4335)$$

$$= 22.3 \text{ p.s.i.g.}$$

Pressure drop at right angled bend (1)

$$= P3 - (\Delta P_t \times 2.6191) / (0.4335 \times \Delta P_t \times D)$$

$$= 111 \text{ diameters of total pressure drop } (\Delta P_t).$$

Pressure drop at right angled bend (2)

$$= (ODP_t - (\Delta P_t \times 4.7125)) / (0.4335 \times \Delta P_t \times D \times B)$$

$$= 125 \text{ diameters of total pressure drop } (\Delta P_t)$$

$$\text{Slip} = \frac{(1 - e_t) - (1 - e_d) \times 100}{e_t (1 - e_d)} \% = 46.8\%$$

Note. The first half of the first page of this appendix lists the data of this run. The second half of that page and the first half of the second page lists the data provided in the computer programme which are common to varying numbers of runs and explicit for this run. The rest of the appendix lists operations in the computer programme for processing the data.

Appendix 5.Comparison of slip rates by 't' - testing.

Material	Line diam.	Average slip rate	Corrected sum of squares	No. of measurements of slip rate
Ballotini	$\frac{3}{4}$ "	35.7278	151.38	18
Sand	$\frac{3}{4}$ "	28.8586	1208.75	29
Ballotini	$\frac{1}{2}$ "	41.4107	938.39	28
Sand	$\frac{1}{2}$ "	33.9933	3668.29	45

Comparison between	Variance	t	Degrees of freedom	Significance level
$\frac{3}{4}$ "	5.4977	4.164	45	0.1%
$\frac{1}{2}$ "	8.055	3.826	71	0.1%
sand	8.2302	2.620	72	2%
ballotini	4.9767	3.780	44	0.1%

Variance is:- (Difference of the sum of squares/degrees of freedom) $^{\frac{1}{2}}$.

't' is:- (Difference of the means)($N_1 \times N_2$)/($N_1 + N_2$) $^{\frac{1}{2}}$ /

Variance where N_1 and N_2 are the number of measurements of the two elements of the comparison.

Degrees of freedom are $N_1 + N_2 - 2$.

If the slip values for the two materials are affected by the extreme opposite estimated errors in determining the free-settled voidage, t becomes 2.892 for the $\frac{3}{4}$ inch line and 2.561 for the $\frac{1}{2}$ inch line. These results are significant to slightly less and slightly more than 1%, respectively.