325
6-14-65

IDO-14648
March 1965

MASTER

SOLIDS FLOW CONTROL FOR
A PULSED SOLIDS-LIQUID CONTACTOR

Earl S. Grimmett and Noel de Nevers

PATENT CLEARANCE OBTAINED. RELEASE TO
THE PUBLIC IS APPROVED. PROCEDURES
ARE ON FILE IN THE RECEIVING SECTION.

**PHILLIPS
PETROLEUM
COMPANY**

ATOMIC ENERGY DIVISION

**NATIONAL REACTOR TESTING STATION
US ATOMIC ENERGY COMMISSION**

DISCLAIMER

This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency Thereof, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

DISCLAIMER

Portions of this document may be illegible in electronic image products. Images are produced from the best available original document.

PRINTED IN USA. PRICE \$3.00. AVAILABLE FROM THE CLEARINGHOUSE FOR FEDERAL
SCIENTIFIC AND TECHNICAL INFORMATION, NATIONAL BUREAU OF STANDARDS,
U. S. DEPARTMENT OF COMMERCE, SPRINGFIELD, VIRGINIA

LEGAL NOTICE

This report was prepared as an account of Government sponsored work. Neither the United States, nor the Commission, nor any person acting on behalf of the Commission:

A. Makes any warranty or representation, express or implied, with respect to the accuracy, completeness, or usefulness of the information contained in this report, or that the use of any information, apparatus, method, or process disclosed in this report may not infringe privately owned rights; or

B. Assumes any liabilities with respect to the use of, or for damages resulting from the use of any information, apparatus, method, or process disclosed in this report.

As used in the above, "person acting on behalf of the Commission" includes any employee or contractor of the Commission, or employee of such contractor, to the extent that such employee or contractor of the Commission, or employee of such contractor prepares, disseminates, or provides access to, any information pursuant to his employment or contract with the Commission, or his employment with such contractor.

IDO-14648
AEC Research and Development Report
Engineering and Equipment
TID-4500 (37th Ed.)
Issued: March 1965

**SOLIDS FLOW CONTROL FOR
A PULSED SOLIDS-LIQUID CONTACTOR**

by

Earl S. Grimmett
Noel de Nevers*

* Faculty member of University of Utah,
Department of Chemical Engineering

PHILLIPS
PETROLEUM
COMPANY

Atomic Energy Division

Contract AT(10-1)-205

Idaho Operations Office

U. S. ATOMIC ENERGY COMMISSION

ABSTRACT

A solids transport and control system has been developed for a pulsed solids-liquid contactor. The system consists of a constant-flow-rate slurry feeder to feed the solids into the contactor and a constant-inventory slurry feeder to withdraw the solids from the contactor, both of which employ a fluidized bed for introduction of solids and a slurry gas lift to move the solids from the bottom of one contactor to the top of another. The entire system has been operated satisfactorily over a wide range of conditions circulating Dowex-50 ion-exchange resin.

SOLIDS FLOW CONTROL FOR
A PULSED SOLIDS-LIQUID CONTACTOR

CONTENTS

ABSTRACT	ii
I. SUMMARY	1
II. INTRODUCTION	2
III. FEED FLOW SYSTEM	5
IV. BOTTOM FLOW SYSTEM	7
V. PERFORMANCE OF COMPLETE CONTACTOR-CONTROLS SYSTEM	8
1. LIMITATIONS IMPOSED BY SOLIDS-LIQUID CONTACTOR	8
2. LIMITATION IMPOSED BY THE BOTTOM DOWNCOMER	8
3. INTERACTIONS BETWEEN CONTROL SYSTEMS AND CONTACTOR	9
4. AIR LIFT PERFORMANCE	10
5. OPERATION WITH ALUMINA CALCINE	10
6. ATTRITION	10
VI. CONCLUSIONS	11
VII. NOMENCLATURE	12
VIII. REFERENCES	13
APPENDIX A -- FEED FLOW SYSTEM	15
1. MECHANICAL DETAILS	17
1.1 Fluidized Bed	17
1.2 Control Instruments	17
1.3 Drawoff Line	19
1.4 Solids Processed	19
2. SYSTEM PERFORMANCE	20
2.1 Solids-Liquid Flow Rates	20
2.2 Solids-to-Liquid Ratio	23

2.3 Air in Lines	24
2.4 Maximum Operable Density	25
2.5 Trapping of Debris	25
APPENDIX B -- BOTTOM FLOW SYSTEM	27
1. CALCULATION OF PRESSURE DIFFERENCE	29
2. MECHANICAL DETAILS	29
2.1 Fluidized Bed	29
2.2 Control Instruments	30
2.3 Valve Sizing	30
3. SYSTEM PERFORMANCE	30
3.1 Pitot-Tube Effect	31
3.2 Trapped Air in Differential-Pressure Cell	31
3.3 Pulsations in Controller Output	32
3.4 Air in Control Lines	32
3.5 Rate Indication	33
3.6 Solids Bypassing	33
3.7 Bed Level	34
APPENDIX C -- SLURRY AIR LIFT TESTS	35
1. PERFORMANCE OF 1/4-INCH PIPE AIR LIFT	37
2. COMPARISON WITH PUBLISHED CORRELATIONS	37
3. BENCH-SCALE SLURRY GAS LIFTS	40
APPENDIX D -- RECOMMENDATIONS FOR FUTURE WORK	45
1. VALVE DEVELOPMENT	47
2. LARGER DOWNCOMER FROM BOTTOM PLATE	48
3. CALCINE OPERATION	48
4. BYPASSING STUDY	48
5. SMALL DIAMETER FEED FLOW FLUIDIZED BED	48
6. CLOSED FEED FLOW SYSTEM LINE	49
7. AIR FLUIDIZED FEED FLOW SYSTEM	49
8. PRESSURE FEED FLOW SYSTEM	49
9. ELIMINATION OF MECHANICAL PULSER	49
10. SLURRY GAS LIFTS	49

FIGURES

1. Simplified flow diagram of pulsed solids-liquid contactor with solids flow systems	3
2. Feed flow system	5
3. Flow diagram of bottom flow system	7
A-1. Modification of research controls motor valve	17
A-2. "Y" style control valve, available in one inch or larger sizes	18
A-3. Solids flow rates through feed flow system (Dowex-50)	21
A-4. Water flow through feed flow system	21
A-5. Solids-liquid ratio of slurry passed by flow system	24
A-6. Solids feeder -- relation of solids content of slurry passing valve to calculated slurry density	24
B-1. Cross sectional view of differential pressure transducer	31
B-2. Effect of pulser frequency on apparent Δp across inventory leg . . .	32
C-1. One-fourth-inch pipe gas lift	37
C-2. Gas lift performance of 14-1/4 feet of vertical 1/4-inch IPS stainless pipe	38
C-3. Gas lift performance of 1/4-inch IPS pipe, 14-1/4 feet long	38
C-4. Comparison of 1/4-inch IPS gas lift with correlation of Chamberlain and Paige ^[5]	39
C-5. Comparison of 1/4-inch IPS gas lift performance with correlation of Govier et al ^[3]	39
C-6. Bench-scale gas lift tester	40
C-7. Performance of 7-mm-ID gas lift for air-water system	41
C-8. Performance of 7-mm-ID gas lift with slurry of Dowex-50 resin in water. Submergence = 0.62	42
C-9. Performance of 7-mm-ID gas lift with slurry of alumina calcine in water. Submergence = 0.62	42
C-10. Performance of 7-mm-ID gas lift with slurry of Dowex-50 resin in water. Submergence = 0.37	43

C-11. Air lift flow reduction due to solids	43
D-1. Valve stems and seats after use	47

TABLES

I. Range of Conditions Over Which Contactor-Controls System was Operated, Circulating Dowex-50	8
A-I. Physical Properties of Solids Processed in Pulsed Solids-Liquid Contactor	20

IDO-14648

SOLIDS FLOW CONTROL FOR
A PULSED SOLIDS-LIQUID CONTACTOR

ADDENDA

Page 18 - Fig. A-2 is reproduced by permission of Hammel-Dahl/Foster
Division, ITT General Controls Inc.

Page 31 - Fig. B-1 is reproduced by permission of Honeywell Inc.

I. SUMMARY

Prior work [1] at this laboratory led to the development of a continuous, pulsed, solids-liquid contactor. However, little attention was then given to the problems of continuously feeding and metering solids into the column, and of removing them from the column. This report describes an experimental study of these problems.

The feed flow system developed during this study receives solids from a slurry gas lift or a solids makeup system and feeds them to the top of the solids-liquid contactor. The system utilizes a bed of solids, fluidized with water or some other process fluid, which is maintained at a constant density by regulating the flow of the fluidizing liquid. The constant density bed is withdrawn at a constant volumetric rate and is introduced into the top of the contactor. Using this system, it was possible to feed Dowex-50 ion-exchange resin into the contactor at controlled rates over a wide range of resin flow rates.

The bottom flow system removes solids from the bottom of the contactor and transports them by a gas lift to the top of the same contactor, to the top of a second contactor, or to the next piece of equipment in a process. The bottom flow system developed in this study also uses a fluidized bed. This bed receives solids from the downcomer of the bottom plate of the contactor and discharges them into a slurry gas lift line. The solids inventory in the bed is controlled by a system which measures this inventory and regulates the solids outflow so that the inventory remains constant. This system was also tested with Dowex-50 resin over a wide range of solid feed rates and contactor pulse frequencies and amplitudes. Under all test conditions, the system functioned smoothly and met the established performance criteria.

A study was made of the performance of a 1/4-inch pipe air lift and of 4.1-mm and 7-mm ID glass tubing slurry air lifts. Slurries were transported successfully in these small diameter lifts, but the performance data could not be correlated with those from published work [6,7] based on larger diameter lifts.

II. INTRODUCTION.

In a previous report, Grimmett and Brown have described the operating characteristics of a pulsed solids-liquid contactor [1]. This device has many possible applications in the nuclear processing field; for example, continuous ion-exchange operations, continuous leaching of solids, purification by crystallization, etc..

Previous work has concentrated on developing the contactor itself, with little attention to the problems of getting solids into and out of it. This report describes an experimental study of the problems of the solids flow, transport, and control accessory to such a contactor. The liquid flow presents no special problems; it was considered only as it affected the solids flow.

The entire system is intended to operate in a hot cell or other radiation environment, where direct maintenance is difficult and costly. Therefore, the solids handling system should:

- (1) Be highly reliable.
- (2) Involve a minimum of moving and/or wearing parts..
- (3) Be as simple as possible.

For a calcine leaching operation the fresh calcine would be delivered to a feed tank above the contactor, flow down through the contactor, and then be removed to another elevated tank for future processing and/or storage. For an ion-exchange operation there would be two pulsed solids-liquid contactors. Stripped resin would flow down one contactor, countercurrent to the fluid from which an ion was to be removed. From the base of this contactor the resin would be lifted to the top of a second contactor, in which it would flow downward, countercurrent to a stripping fluid. The stripped resin would then be returned to the top of the first contactor, completing the cycle.

The solids transport and flow control problems for the systems described above are:

- (1) To introduce the solid, from some elevated feed tank, into the top of the contactor at a steady, measured rate, which may be varied over some range.
- (2) To remove the solids from the bottom of the contactor and insert them into the stream or device which transports the solids to their next processing step.
- (3) To transport the solids to their next processing step..

Figure 1 shows the simplified flow diagram of the test loop used for this study. The loop consists of a two-inch ID pulsed solids-liquid contactor, a feed flow system for introducing the solids into the top of the contactor, a bottom flow system for removing solids from the bottom of the contactor, and a slurry air lift for returning the solids to storage above the contactor and

Fig. 1 Simplified flow diagram of pulsed solids-liquid contactor with solids flow systems.

thence into the feed flow system. These subsystems are described in detail in subsequent sections of this report.

This entire loop has only three moving parts, two pneumatic control valves and the piston pulser for the contactor. If necessary, for remote operation, the piston pulser might be replaced with a pneumatically driven pulser, thus eliminating one of these moving parts.

The loop has been operated successfully using Dowex-50 ion-exchange resin as the solid, and water as the fluid, over a wide range of feed rates, pulser frequencies, and amplitudes. Scoping operations with calcined alumina were more difficult because the small valves used in the system were easily plugged; however, changes in the design of these valves should make it possible to operate the system with calcined alumina.

III. FEED FLOW SYSTEM

The feed flow system receives solids from the slurry gas lift or the make-up system and feeds solids into the top of the contactor. For the processes envisaged here, the solids will be delivered as a slurry, so that the feed flow system must handle wet solids.

The requirements for this system are:

- (1) It should add solids to the contactor at a steady rate.
- (2) It should remotely indicate this rate, or have this rate subject to a reproducible calibration in the terms of variables remotely indicated.
- (3) The range of rates available should be large; change from one rate to another should be easy.
- (4) It should be simple, reliable, and trouble-free.

The operating characteristics of the fluidized bed makes the feed flow system possible. The slurry of solids is fluidized with water or some other process fluid. This fluidized bed is held at constant density (ie, constant solids content) by a controlled flow of fluidizing water. A drawoff line withdraws a steady volume flow rate of this bed and introduces it into the contactor. The volume (and, hence, mass) rate of solids flow to the contactor is controlled by regulating the fluidized bed density and the drawoff rate. (In this pilot loop the steady volume flow rate was obtained by using an on-off flow for reasons discussed in Appendix A.)

Figure 2 shows this system. The slurry of solids and transport fluid enters the top of the inventory section from the air-separator cyclone of the slurry gas lift. In the inventory section (six-inch ID) the upward water velocity is less than the minimum velocity required to fluidize the solids; the solids settle to form a packed bed in the bottom and neck of the inventory section. Below the inventory section is the fluidized bed section (two-inch ID). The same fluid which flows up through the inventory section first flows up through the fluidized bed section. However, here, because of the reduction of diameter, its upward velocity is nine times as great as in the inventory section. This

Fig. 2 Feed flow system.

velocity is sufficient to produce the desired fluidized bed below the packed bed in the inventory section.

The mechanical details of this system are discussed in Appendix A.

IV. BOTTOM FLOW SYSTEM

The bottom flow system allows solids to flow out of the contactor into the slurry gas lift line. The requirements for this control system are:

- (1) It should remove the solids from the contactor at whatever rate they flow down the downcomer from the lowest plate. (This rate fluctuates.)
- (2) It should allow as little fluid as possible to flow out with the solids.
- (3) Its operation should be smooth and should not upset the operation of the contactor.
- (4) It should be simple, reliable, and trouble-free.

The bottom flow system also uses a fluidized bed. This fluidized bed receives solids from the downcomer of the bottom plate of the contactor. It discharges solids into the slurry gas lift line. The solids inventory in the bed is controlled by a system which measures this inventory and regulates the solids outflow to hold this solids inventory constant.

Figure 3 shows this system. The fluidized bed is formed in a piece of 1/2-inch ID glass pipe. A small, steady flow of water up the pipe maintains the solids in the dense-phase fluidized state. Two pressure taps, three feet apart, sense the difference in pressure between the top and bottom of the bed. This pressure difference is transmitted through water-filled lines to the differential pressure transducer. It is shown in Appendix B that the pressure difference observed by this differential pressure transducer is directly proportional to the mass of solids contained in the bed region between the two pressure taps. Thus, holding this differential pressure constant maintains a constant solids inventory.

The mechanical details of this system are discussed in Appendix B.

Fig. 3 Flow diagram of bottom flow system.

V. PERFORMANCE OF COMPLETE CONTACTOR-CONTROLS SYSTEM

The entire system -- contactor and controls -- was run continuously as an integral unit without control or solids transport difficulties over a wide range of feed rates, pulse amplitudes, and frequencies. Table I shows the range of operating conditions under which the integral unit was run.

TABLE I

RANGE OF CONDITIONS OVER WHICH CONTACTOR-CONTROLS
SYSTEM WAS OPERATED, CIRCULATING DOWEX-50

Pulser amplitude ^[a]	1/16, 1/8, and 1/4 inch
Pulser frequency ^[b]	40 to 2930 cycles/min
Solids flow rate ^[c]	8 to 20 g/min = 48 to 121 lb/hr ft ²
Liquid flow rate up contactor ^[d]	0.002 to 0.112 gpm = 47 to 2630 lb/hr ft ²
Interval between solid additions ^[e]	15 to 115 sec

- [a] No lower limit observed; upper limit set by violent mechanical shaking of contactor for 1/4-inch stroke and pulser frequencies above 70 cycles/min. Pulser piston OD is the same as contactor ID so a given pulser stroke causes a corresponding movement of fluid in the contactor.
- [b] Lower limit is slowest rate at which pulser motor functions without stalling. No upper limit was observed.
- [c] Upper limit discussed in text; no observed lower limit.
- [d] Limits set by size of water rotameter.
- [e] Limits set by convenient settings of Flexopulse timer.

1. LIMITATIONS IMPOSED BY SOLIDS-LIQUID CONTACTOR

As discussed in a previous report [1], there are some pulse frequencies (about 700 to 900 cpm) at which the solids do not form a coherent bed on the plates of the contactor. When the system was tested at these frequencies the contactor acted merely as a delay in the flow of solids from the feed flow system to the bottom flow system. The control systems worked properly. Thus, these pulser frequencies represent a limitation on the contacting efficiency of the system, but not on the solids transport and control devices.

2. LIMITATION IMPOSED BY THE BOTTOM DOWNCOMER

The maximum flow of Dowex-50 ion-exchange resin through this test system was about 15 to 20 g/min. This limit was set by the maximum flow rate

possible through the downcomer leaving the bottom stage of the contactor, a rate which was considerably less than the rates possible through the other downcomers in the column. The reason for the lower rate in this downcomer is that the net upward flow of liquid through it is higher than through the other downcomers. The limitation of solids flow set by the bottom downcomer need not exist in a well designed column. It is possible to increase the diameter of the bottom downcomer so that the flow rate of solids through it matches the flow rate of the solids through the other downcomers in the system.

3. INTERACTIONS BETWEEN CONTROL SYSTEMS AND CONTACTOR

Except for the flow rate limitation through the bottom downcomer, no interactions between the contactor and the bottom control system were observed. For a constant-inventory controller setpoint the bottom flow system maintained approximately the same level of dense-phase fluidized bed in its inventory leg.

Interactions between the solids feed flow system at the top of the column, and the contactor were apparent, however. The "normal" mode of operating the feed system was one in which the closed periods of the solids feed valve were as short as convenient, eg, about 15 seconds. However, closed periods as long as 115 seconds (longest possible with a Flexopulse) were successfully used. When these long periods with no flow are used, the solids-addition pulse is visible, moving down through the column. When the valve opens, the fluidized bed level on the top plate begins to rise. Soon the flow down the downcomer from the first plate increases, and the second plate fluidized bed level begins to rise. Meanwhile the solids addition pulse has ended, and the level on the first plate falls to the level of the top of its downcomer. In a similar fashion the "pulse" of extra solids passes from plate to plate, reaching the bottom (fifth) plate 40 to 50 seconds after the valve opened. This delay time is not markedly changed by large changes in pulser frequency.

On the bottom plate two types of behavior may occur. If the total solids flow, averaged over the time interval, is close to the maximum which the downcomer from the bottom plate will pass, then the level on the bottom plate will fall slowly, and be near its lowest value, when the next pulse arrives. Under these conditions the solids flow rate to the bottom flow controller is roughly constant. The other type of behavior occurs when the solids flow rate, averaged over the time interval, is significantly less than the maximum that can pass down the bottom downcomer. In this case the level on the bottom tray will fall to the level of the top of its downcomer, then flow down the downcomer will practically cease until the next pulse arrives. In this case the flow to the bottom flow controller will be on-off.

It is possible that this "pulsed solids flow" mode of operation may give better mass transfer efficiency than the quasi-steady flow obtained with shorter valve-closed times. McWhirter and Lloyd [2] have shown that for distillation columns a somewhat analogous flow situation gives better mass transfer efficiency than steady flow. Determination of this mass transfer performance was beyond the scope of this study. Under all these conditions, the control systems functioned without difficulty, and the contactor maintained smooth, even fluidized beds on every plate.

The feed flow controller discharged its slurry through an air gap of several inches into a pool of liquid in the contactor's feed funnel. This isolated it from the pulsations in the column and made it easy to calibrate.

4. AIR LIFT PERFORMANCE

As shown in Figure 1, the solids were transported from the bottom solids flow system to the solids feed system at the top of the column by a slurry air lift. Although this lift system functioned smoothly and presented no particular problems, a brief study on these systems was initiated because of the absence of published information on the operation of small diameter air lifts and slurry air lifts.

Data were obtained from a 1/4-inch-diameter pipe air lift, and from a 4.1-mm and a 7-mm ID glass tubing slurry air lift. The results obtained from the 1/4-inch pipe air lift study were not what would be predicted from published correlations [5] of data from larger diameter gas lifts. The results obtained from the slurry air lift study indicated that the effect of substituting a slurry for a pure liquid is simply an increase in the viscosity of the fluid being pumped. Appendix C summarizes the results of these studies.

5. OPERATION WITH ALUMINA CALCINE

After the system had been successfully operated with Dowex-50 water slurries, it was tested with slurries of alumina calcine in water. These were more difficult to handle because they readily plug the small diameter valves used here.

6. ATTRITION

The resin suffered measurable attrition in passing through the system. The resin was originally all in the size range -22 +32 Tyler screen size (Table A-I). One batch of resin which was circulated through the system an average of about 30 times was 12 weight percent smaller than 32 mesh. Probably most of this attrition occurred in the valves. A better valve design should reduce this attrition (see Appendices A and D).

When the system was operated at high flow rates, the fines accumulated in the inventory section of the feed flow system. At low flow rates the fines were more evenly distributed, accumulating also on the tops of the fluidized beds on the various plates and on the fluidized bed in the bottom flow system. These fines caused no control or transport problems.

VI. CONCLUSIONS

Recommendations for further studies of possible improvements on the solids feed system for the continuous solids-liquid contactor are summarized in Appendix D. As a result of the present study, the following conclusions have been made.

- (1) Fluidized beds can be used for constant-solids-flow-rate and constant-solids-inventory controls. These control systems are simple and reliable, each requiring only one mechanical device. This simplicity makes them attractive for use in remote processing of radioactive materials.
- (2) Slurry gas lifts are simple, reliable devices for transporting wet solids vertically and horizontally.
- (3) A complete controls-contactor system has been demonstrated which contacts fluids countercurrent to a recirculating mass of granular solids, using only parts suitable for remote operations.

VII. NOMENCLATURE

D	Pipe diameter	ft
f'	Modified friction factor used by Brown et al ^[3]	dimensionless
g	Acceleration of gravity	32 ft/sec ²
g _c	Force-mass conversion factor	32 lb _m ft/lb _f sec ²
Δh	Difference in height between pressure taps or ends of dip tubes	ft
Δp, Δp _{gross} , Δp _{net}	pressure differences	lb/in. ²
S	Submergence	dimensionless
U _g	Superficial velocity of gas	ft/sec
U _l	Superficial velocity of liquid	ft/sec
V _a	Volume flow rate of air	ft ³ /min
V _l	Volume flow rate of liquid	ft ³ /min
ρ _g	Density of gas	lb _m /ft ³
ρ _f	Density of fluid	lb _m /ft ³
ρ _s	Density of solid particles	lb _m /ft ³
ρ _{avg}	Average density of fluidized bed	lb _m /ft ³

VIII. REFERENCES

1. E. S. Grimmer and B. P. Brown, "A Pulsed Column For Countercurrent Liquid-Solids Flow", Ind. Eng. Chem., 54 (November 1962) pp 24-28.
2. J. R. McWhirter and W. A. Lloyd, "New look at distillation. V. Controlled cycling in distillation and extraction", Chem. Eng. Progr., 59 (June 1963) pp 58-63.
3. R. A. S. Brown, G. A. Sullivan, and G. W. Govier, "The upward vertical flow of air-water mixtures. III. Effect of gas-phase density on flow pattern, holdup, and pressure drop", Can. J. Chem. Eng., 38 (1961) pp 62-66.
4. J. H. Perry et al, Chemical Engineer's Handbook, Fourth Edition, McGraw-Hill (1963) pp 5-6.
5. H. V. Chamberlain, "Factors Affecting Air-Lift Performance". Paper presented at meeting, AIChE, Salt Lake City, Utah, September 21-24, 1958.
6. H. V. Chamberlain, Factors Affecting Capacity of Air Lifts, IDO-14398 (March 1957).
7. H. V. Chamberlain, Air Lift Performance at Low Liquid Rates Using Oversized Piping and Lateral Runs, IDO-14561 (October 1961).
8. G. A. Hughmark and B. S. Pressburg, "Holdup and Pressure Drop With Gas-Liquid Flow in a Vertical Pipe". A.I.Ch.E. J., 7 (December 1961) pp 677-682.
9. D. S. Scott, "Properties of Concurrent Gas-Liquid Flow", Advances in Chemical Engineering, V. 4 (1963) pp 199-277.
10. J. H. Perry, Op. cit., pp 3-229.
11. B. P. Brown et al, Development of a Fluidized Bed Calcination Process for Aluminum Nitrate Wastes In a Two-Foot-Square Pilot Plant Calciner; Part I. Equipment Development and Initial Process Studies, IDO-14586 (June 1962).

THIS PAGE
WAS INTENTIONALLY
LEFT BLANK

APPENDIX A

FEED FLOW SYSTEM

THIS PAGE
WAS INTENTIONALLY
LEFT BLANK

APPENDIX A -- FEED FLOW SYSTEM

1. MECHANICAL DETAILS

1.1 Fluidized Bed

The fluidized bed is formed in a section of two-inch ID glass pipe. The density controller dip tubes are set with their ends 20 inches apart. This length is adequate for smooth density control; a taller bed would give a larger signal and, hence, possibly finer control, but this would make the feed flow system taller and thereby increase the overall system height.

The system was successfully operated with a one-inch ID fluidized bed; however, this bed was difficult to control. The flow out the drawoff line was large enough, relative to the fluidizing water flow, to cause a significant interaction between the bottom drawoff and the bed density control. When the solids drawoff line is open, there is a different upward flow velocity at the lower density probe (below the drawoff) than at the upper density probe (above the drawoff). In the two-inch ID bed this change is small compared with the fluidizing water flow, and affects the density control only slightly. In the one-inch ID bed the interaction is significant. Also, the hook on the top of the drawoff line occupied a significant part of the bed cross section in the one-inch ID bed, disturbing the flow and causing the fluidized bed to be visibly nonuniform there. This problem did not exist in the two-inch ID bed.

1.2 Control Instruments

The control valve was a modified Research Controls 1/4-inch IPS Model P755. Figure A-1 shows the original form of this valve and the modified form. The diaphragm, valve positioner, etc, were not modified. The original valve was of the long-taper needle type. For vertical flow the flow path included a horizontal section through the seat, two 120° turns and two 30° turns. The modified valve has a 45° taper stem and a flow path with no horizontal sections: one 90° turn and two 45° turns.

Fig. A-1 Modification of research controls motor valve.

The change in stem type was needed to provide a large enough opening for the solids to pass. The original long-taper needle, in the wide open position, did not have a maximum clearance equal to the ion-exchange resin particle diameter. The 45° taper stem, in the wide open position, has an opening equal to the ID of the seat orifice (0.156 inch).

The change in flow pattern was made to eliminate plugging. The first modification tried, which had only the stem design change mentioned above, plugged

occasionally in ion-exchange resin service. After the flow pattern was modified, plugging with ion-exchange resin never occurred. The valve was constructed at the NRTS because such a valve is not commercially available in small sizes. However, such valves are available commercially in sizes above one inch (Figure A-2).

Fig. A-2 "Y" style control valve, available in one inch or larger sizes.

After modification, the valve behaved as an on-off valve. With slurry in the valve, as the valve slowly opened from its closed position, the sequence of events was:

- (1) As the stem lifted off the seat, water began to flow through. However, since the opening was still smaller than the diameter of the particles, no particles passed. Thus the valve acted as a strainer, straining the solids out of the slurry. These solids formed a packed bed in the body of the valve.
- (2) When the valve opening became as large as the particle diameter, the water flowing through the valve began to drag the solids through the seat. The packed bed in the valve body moved downward as the particles below it were removed. At this valve position the water flow rate through the seat was fast enough that the slurry flowing into the valve contained as many particles as were flowing through the valve, so that the packed bed moved downward but re-formed at the top as rapidly as it disappeared at the bottom.
- (3) When the valve opening became so large that solids flowed out faster than the slurry brought them in, the packed bed

in the valve body disappeared, the flow resistance due to this packed bed also disappeared, and the volume flow rate through the valve increased suddenly.

- (4) Further stem movement had little effect on the flow because the principal resistance was the flow resistance of the valve seat.

The entire sequence of events described above occurs in a valve stem movement of about one particle diameter. For the valves tested, which operate on 3 to 15 psi signal pressure handling Dowex-50 water slurries, this movement corresponded to about 0.2 psi. Thus this valve acted as an on-off rather than as a throttling valve.

The valve caused some attrition of the solid particles passing through it. This is discussed in detail in Section V of this report. Recommendations for developing more satisfactory valves are included in Appendix D of this report.

Because of the on-off character of the valve, an on-off controller was used for it. This was a Flexopulse electric timer, actuating a solenoid valve in the air supply line to the control valve diaphragm. With larger systems, it should be possible to design a solids flow valve which does not have this on-off character, and thus obtain a workable steady-flow solids feed system. This on-off feature does not decrease the usefulness of the system used here. However, the valve should have a longer service life in a throttling system than in this on-off system, and the particle attrition in the valve should be less severe.

The fluidized bed density control posed no unusual hardware problems. The instruments used were a Honeywell Model N-7 differential pressure transducer set for a range of 0 to 4 inches of water, a Foxboro Model 58P5 controller, and a Hammel-Dahl "Y" style split body control valve.

1.3 Drawoff Line

The first drawoff line tried was simply a vertical tube open at the top. This was unsatisfactory because, in addition to drawing off part of the fluidized bed when the valve was open, it also collected solids which entered its mouth by free settling when the valve was closed. Sometimes the flow currents in the bed pointed downward in the neighborhood of its mouth, and the tube filled completely with resin, and formed a small packed bed standing on top of the tube. Under these circumstances the amount of solid and liquid flow through the valve when it was open was not a reproducible function of bed density and pulser settings.

The problem was remedied by making a hook-shaped drawoff line with the inlet pointing downward. With this drawoff no measurable quantity of solids entered the tube by free settling (regardless of the flow currents in the fluidized bed) and the flows were a reproducible function of bed density and timer setting.

1.4 Solids Processed

Most of the solids transport and control development work for this project was done with Dowex-50 ion-exchange resin as the solid being processed. Some tests were also made with an alumina calcine, typical of that prepared in the

pilot plant for the Idaho Waste Calcining Facility [11]. The pertinent physical properties of these materials are summarized in Table A-I.

TABLE A-I
PHYSICAL PROPERTIES OF SOLIDS
PROCESSED IN PULSED SOLIDS-LIQUID CONTACTOR

	Resin	Calcine
True Density of Dry Solid Particles (g/cc)	$\approx 1.3^{[a]}$	≈ 2.3
Porosity of Individual Solid Particles	25% ^[a]	40 to 50%
Dry Solids Content of a Settled Bed of Solids in Water (g/cc) (This was determined to permit volume measurements in a graduated cylinder to substitute for dry solids measurements.)	0.47	1.15
Hardness (diamond pyramid hardness)	17 to 21	130 to 160
Screen Analysis (wt%)		
Tyler Screen Size 16	----	0.8
20	----	6.8
24	11.8	7.4
28	45.8	29.9
32	42.4	17.2
35	----	11.8
48	----	21.7
65	----	4.1
100	----	0.3
	100.0	100.0

[a] These values vary with the ion content of the resin and are only approximate.

2. SYSTEM PERFORMANCE

2.1 Solids-Liquid Flow Rates

As mentioned before, the solids flow valve functions only as an on-off valve, not as a throttling valve; it was controlled by a Flexopulse timer, which opened and closed a solenoid valve in the air supply line to the motor valve's diaphragm. The Flexopulse timer has two settings -- length of open period per cycle and length of closed period per cycle. Therefore, there were three independent controller settings for this system, the bed density (as set by the

density controller) and the open and closed periods of the Flexopulse timer. The solids and liquid flow rates for Dowex-50 passing through the system are shown as a function of these variables on Figures A-3 and -4, respectively.

Fig. A-3 Solids flow rates through feed flow system (Dowex-50).

Fig. A-4 Water flow through feed flow system.

Figure A-3 shows that:

- (1) Increasing the length of time the valve is open per pulse increases the mass of solids passed per pulse; this increase is practically linear.
- (2) At low solids contents of the bed, increasing the time the valve is closed between pulses decreases the mass of solids passed per pulse, but at higher solids contents in the bed this effect diminishes.
- (3) Increasing the bed solids content causes the solids passed per pulse to increase in the solids content range from 10 to about 17 weight percent. Further increase in the solids content of the bed cause the solids passed per pulse to decrease sharply in the 17 to 22 weight percent solids range. At higher solids contents in the bed, increasing the solids content causes the solids passed per pulse to decrease slowly.

Figure A-4 shows that:

- (1) Increasing the length of time the valve is open per pulse increases the liquid flow per pulse; this increase is practically linear.

- (2) The effect on liquid flow of increasing the length of time the valve is closed between pulses is small. At high densities the liquid flow per pulse is highest for long periods (10 sec) between pulses.
- (3) Increasing the solids content of the bed causes the liquid passed per pulse to decrease slowly in the 10 to 17 weight percent range, rapidly in the 17 to 22 weight percent range, and slowly in the 22 weight percent plus range.

These observations can be explained as follows:

(1) The linear increase in flow of solids and liquids per pulse with increased length of time the valve is open is self-explanatory; the flow per second is roughly constant. If more precise data were available it would probably be observed that the increase of flow-per-pulse with open time was linear, with a slight offset from zero-flow at zero-time as shown in the following sketch:

HYPOTHETICAL FLOW BEHAVIOR
AS A FUNCTION OF OPEN TIME

This would be caused by the time required to start the flow of solids. When the valve opens, the flow starts slowly, quickly increases to a maximum value, and then remains constant. (This start-up takes roughly 0.2 second; it is the time required for gravity to accelerate the fluid to its terminal flow velocity. The maximum velocity in the outlet tube was about five feet per second. A body falling freely from rest requires about 0.15 second to reach this velocity.) However, the low-open-time data are not precise enough to show this hypothetical behavior. The accuracy of setting the Flexopulse is only about plus or minus 0.1 second; this imprecision in the flow data is therefore not surprising.

(2) The decreased solids flow with increased time between pulses is probably due to settling in the solids line. When the valve closes, the solids line above it is full of slurry of the same composition as the fluidized bed. In the valve-closed period, some of the solids settle to the bottom of the line and form a packed bed. When the valve opens, this packed bed restricts the flow until it is washed away. The longer the valve is shut between openings the more this effect should retard the flow.

Presumably, there is a maximum period after which all the solids in the drawoff line will have settled. After that period, lengthening the valve-closed period should not affect the flow. If this maximum period exists, it is longer than 10 seconds (the maximum valve-closed period used here).

(3) With increasing solids content in the slurry, the viscosity of the slurry increases. More of the flow resistance is viscous resistance in the lines, and less is inertial resistance in the valve. This explains the decreased effect of valve-closed time on solids flow as bed solids content increases. As the flow becomes more viscous, the packed bed formed at the valve has less effect on the flow.

(4) The maximum in each curve in Figure A-3 is due to two conflicting effects. Increasing the weight percent solids in the fluidized bed increases the viscosity of the slurry. As shown in Figure A-4 this results in a continual decrease in water flow rate with increasing solids content of the bed.

At low solids contents, the weight percent solids in the bed increases faster than the total flow rate decreases, so that the mass of solids passed per pulse increases with increasing solids content. At high solids contents, the total flow decreases faster than the weight percent solids increases, so that the mass of solids passed per pulse decreases with increasing solids content.

(5) When shut off, the solids feed valve leaked water at a rate of about 60 ml/min. This explains the increase in water flow with increasing valve-closed periods. The leakage during the closed periods contributed to the total liquid flow and made the total flow per cycle larger.

2.2 Solids-to-Liquid Ratio

In the foregoing section the weight percent solids reported are based on the readings of the bed density controller. This controller measures the difference in pressure between two points in the bed 20 inches apart vertically, with dip legs filled with water; the instrument is ranged to have full-scale reading when the differential pressure is four inches of water. The weight percent solids is calculated from

$$\text{wt\% solids} = \frac{\Delta p}{\frac{(\rho_s - \rho_f)}{\rho_f} \Delta h \cdot g} \quad (1)$$

Here Δp is the pressure differential indicated by the density controller, ρ_s is the density of the solid particles, ρ_f is the density of the liquid, Δh is the vertical distance between pressure dip tube ends, and g is the acceleration of gravity.

Figure A-5 shows the measured g/pulse of solids versus g/pulse of liquid for three different density controller settings. These data scatter somewhat, but they can be represented approximately by the straight lines shown. Each of these lines corresponds to a constant weight percent solids. Similar plots were made for all the experimental density controller settings. The weight percent solids determined by the solids-liquid ratio flowing through the valve as in Figure A-5 is compared with the weight percent solids calculated from the density controller reading, in Figure A-6. The agreement between the two values of weight percent solids is fair. Note that the results seem to lie on two different lines, and that these lines correspond to the high-liquid-flow-rate and low-liquid-flow-rate parts of the liquid flow rate versus density curves. This suggests an interaction between the fluid flow in the fluidized bed section and the density controller. The cause is probably the fact that the upper (low pressure)

Fig. A-5 Solids-liquid ratio of slurry passed by flow system.

Fig. A-6 Solids feeder -- relation of solids content of slurry passing valve to calculated slurry density.

probe is above the drawoff line, and the lower probe is below it. Thus, since there is a discontinuity in vertical flow velocity at the drawoff line, each probe is in a different flow field.

The primary requirement of the control system is that it gives reproducible flow rates for given controller settings. This function is not impaired by this interaction of the flow and the density controller, as long as the system is treated purely as a calibrated system. These results do indicate that the observed performance of a new system may differ from its designer's prediction, and that each system should be calibrated.

2.3 Air in Lines

The pernicious effects of trapped air bubbles on this control system can

hardly be overemphasized. When tap water was used for fluidizing and for the density probes, air bubbles accumulated in various places causing control malfunctions. Even using demineralized water, which is undersaturated with respect to air at room temperature, there were occasional bubbles in the density controller dip tubes, which required flushing of these lines.

Every time the system was drained (and thus filled with air) the drawoff line trapped a bubble of air in its high point. This bubble could seldom be

removed by opening the solids feed valve and expecting the liquid in the column to flush the bubble out. Instead it was often necessary to back flush it out with water from a temporary water line.

This problem should be much less troublesome in larger-size equipment. The problem was much more severe in a one-inch ID column than it was in a two-inch ID column.

2.4 Maximum Operable Density

It is desirable to operate this feed system at the highest possible weight percent solids. This minimizes the volume of water flowing out with the solids and, also, minimizes the volume of water required to fluidize the solids. In cases where leaching of the solids is to be controlled, this latter factor may be significant.

In the two-inch ID fluidized bed, the maximum operable density was about 30 weight percent Dowex-50. At higher densities, the bed could be controlled only if the solids drawoff valve were not opening. When this valve opened, the net flow through it was large enough to reverse the water flow direction in the upper part of the bed, making the net water flow downward. In this situation, the direction of the pressure gradient reversed, and the density signal dropped rapidly. This shut off the fluidizing water flow, and the entire bed transformed from a fluidized bed to a packed bed. Once this occurred, the weight of the resin in the bed was supported by the bottom of the fluidized bed column, rather than by the fluid flowing upward; thus, the density controller could not function.

The maximum operable density in the one-inch ID fluidized bed was about 23 weight percent Dowex-50. The narrower bed was inherently more difficult to control and went through the loss-of-fluidization described above at 23 weight percent Dowex-50. From this it appears likely that in a bed larger than two-inch ID the maximum operable density may be higher than the 30 weight percent described above.

The flow diagram for this system (Figure 2) shows a manual bypass around the fluidizing water flow control valve. To operate at high densities, it is necessary to take part of the fluidizing water flow through this bypass. If this is not done, any small upset will cause the density controller to close the water valve completely, and the loss-of-fluidization described above will occur. The amount of water bypassed around the control valve should be the minimum amount which will cause dense-phase fluidization of the bed.

2.5 Trapping of Debris

The section of the fluidized bed below the fluidizing water inlet served as a debris trap. Those foreign particles, heavier than the ion-exchange resin which found their way into the system, accumulated there. In a full-scale unit a drain valve at this point would allow the occasional removal of this debris.

THIS PAGE
WAS INTENTIONALLY
LEFT BLANK

APPENDIX B

BOTTOM FLOW SYSTEM

THIS PAGE
WAS INTENTIONALLY
LEFT BLANK

APPENDIX B -- BOTTOM FLOW SYSTEM

1. CALCULATION OF PRESSURE DIFFERENCE

The gross pressure difference between the two pressure taps (Figure 3) is

$$\Delta p_{\text{gross}} = \rho_{\text{average}} g \Delta h . \quad (2)$$

Here Δp_{gross} is the gross pressure differential between the two taps, ρ_{average} is the average density of the slurry between the taps, Δh is the vertical distance between taps, and g is the acceleration of gravity. However, the lines from these pressure taps to the differential pressure transducer are filled with water, and the one from the lower pressure tap has a greater vertical length, by exactly Δh . Therefore, these water-filled lines subtract from the above pressure

$$\Delta p_{\text{lines}} = \rho_{\text{water}} g \Delta h . \quad (3)$$

Here ρ_{water} is the density of water. The net signal seen by the pressure transducer is

$$\Delta p_{\text{net}} = (\rho_{\text{average}} - \rho_{\text{water}}) g \Delta h . \quad (4)$$

The average density of the fluidized bed is

$$\begin{aligned} \rho_{\text{average}} &= (\text{volume fraction solids}) \rho_{\text{solid}} \\ &+ (\text{volume fraction water}) \rho_{\text{water}} . \end{aligned} \quad (5)$$

Because the two volume fractions sum to unity, this can be simplified to

$$\rho_{\text{average}} = \rho_{\text{water}} + (\text{volume fraction solids}) (\rho_{\text{solids}} - \rho_{\text{water}}) . \quad (6)$$

Substituting this value of ρ_{average} into Equation (4) yields

$$\Delta p_{\text{net}} = (\text{volume fraction solids}) (\rho_{\text{solids}} - \rho_{\text{water}}) g \Delta h . \quad (7)$$

Thus, since the distance between taps and the densities are constant, the differential pressure depends only on the volume fraction solids in the measuring leg. Holding this differential pressure constant holds the volume fraction solids, and therefore the solids inventory constant.

2. MECHANICAL DETAILS

2.1 Fluidized Bed

The fluidized bed (Figure 3) is made of 1/2-inch ID glass pipe. The two pressure taps are 1/4-inch OD glass tubes. The lower pressure tap enters and then turns to face vertically downward; the reason for this is discussed later in this section of the report.

2.2 Control Instruments

The differential pressure cell used is a Honeywell Model N-7. The range is 0 to 5 inches water column. This instrument is an old-fashioned, flexible-diaphragm instrument. It was mounted with its diaphragm vertical for reasons described later in this section of the report. A more modern Foxboro Model 15-A instrument with a silicone-oil-filled diaphragm, originally tried in this service, was unsatisfactory. It appears that the massive diaphragm of this instrument interacted with the pulsations in the system causing resulting loss of signal. The controller was a Foxboro Model 58P5.

The control valve was very similar to the control valve described in the section on the Feed Flow System. However, it was modified by drilling a hole into the body directly above the valve seat. The fluidizing water (shown in Figure 3) was introduced through this hole. This minimized the likelihood of plugging in the valve. Earlier versions of the valve plugged occasionally when processing Dowex-50 water slurries; the final form described here did not plug when metering Dowex-50 water slurries.

2.3 Valve Sizing

In selecting a valve for this service the designer is limited by the maximum size particle to be passed through the valve. The diameter of the maximum valve opening must be selected several times as large as this maximum particle diameter. The maximum opening of the valve used here was 0.156 inch. The maximum diameter resin particle was 0.034 inch.

A problem in control of the system is the flow of water downward through the valve. It is desirable that this flow be small compared with the flow of fluidizing water up through the fluidized bed so that opening and closing the valve will not upset the column. However, this fluidizing water flow is limited by the diameter of the downcomer from the bottom plate. The net upward water velocity (with the valve closed) must be smaller than the free settling velocity of the smallest particle in the system, or the small particles will not pass through the downcomer.

Increasing the column diameter will partially alleviate this situation. Thus the same valve size can be used on larger columns and will result in a desirable increase in the ratio of the fluidizing water flow rate to the water flow rate through the valve. For any size column, the control system can be improved by using a larger downcomer from the bottom plate than from the other plates. This sacrifices some column contacting performance, but should improve the control stability.

3. SYSTEM PERFORMANCE

After the system was debugged, it functioned satisfactorily for all ranges of conditions tried. These are summarized in Section V of this report. The solid used was Dowex-50 ion-exchange resin. The control system is capable of operating over a wider range of conditions than is the solids-liquid contactor. Thus, the range of conditions which could be tested was limited by the solids-liquid contactor.

3.1 Pitot-Tube Effect

The system tried first did not contain the downward-facing end on the lower pressure-measuring tap. It was observed that when the downward flow of solids and liquid through the system became large the pressure drop across the system would increase, opening the valve wider still, causing the pressure drop to further increase, etc. This "runaway" of the control system emptied the system of solids and discharged a large amount of liquid from the contactor, upsetting the operation of the contactor.

The apparent cause of this malfunction was that the lower pressure-measuring tap pointed slightly upstream. Thus, when flow started downward, the two pressure taps acted as a pitot tube [4], giving a signal which increased with the flow rate. This amounted to a positive feedback on the controller and led to a runaway. By placing the downward hook on the lower pressure-measuring tap, this pitot-tube effect was reversed; downward flow of the fluid in the tube caused an apparent decrease in the measured pressure differential. This corresponds to a negative feedback on the system controller, and completely eliminates these runaways.

3.2 Trapped Air in Differential-Pressure Cell

Figure B-1 shows a cross section of the differential pressure transducer used to measure the solids content of the constant-inventory leg.

Fig. B-1 Cross sectional view of differential pressure transducer.

The normal mounting for this type instrument is with the diaphragm horizontal. In this position the only bleed for air from under the diaphragm is through the "High Pressure Vent" shown on Figure A-7. Since this vent is below the diaphragm, it cannot remove all the air.

This air, trapped in the instrument, can cause a false measurement of the differential pressure (Δp) across the inventory leg. This Δp is also measurable by a manometer in parallel with the dp cell. Figure B-2 shows this Δp

Fig. B-2 Effect of pulser frequency on apparent Δp across inventory leg.

pulsed condition, no spurious Δp is observed. If the transducer is installed with its diaphragm vertical, so that all the air can be removed, this spurious Δp also disappears. Although this vertical-diaphragm mounting is contrary to common practice with this type of instrument, it has been used successfully here with no ill effects.

An alternate explanation which was explored was that oscillations in the piping system were causing this spurious Δp . The computed natural frequencies of oscillation of the pipe for longitudinal oscillations are about 27,000 cycles/minute, and for radial oscillations several million cycles/minute. Clearly such oscillations are not excited by the low pulse frequencies (up to 2930 cycles/minute) used here.

3.3 Pulsations in Controller Output

Under all circumstances the output pressure of the controller oscillates over a short range. This is presumably due to the on-off characteristic of the modified control valve. Most of its flow regulation occurs over a very narrow range of controller output pressure. These pulsations do not upset the operation of the contactor, nor the control system.

3.4 Air in Control Lines

In the early stages of the test program, it appeared that some of the operating troubles were caused by air bubbles which were visible in the system. Since laboratory tap water is supersaturated with air, it releases bubbles in equipment. This problem was partially solved by using demineralized water for the fluidizing water stream. The demineralized water is not supersaturated with air.

Later, after other operating deficiencies had been found and removed, it appeared that bubbles in the system were of minor importance. Stable operation was observed for several half-hour intervals with a large air bubble trapped in

the fluidized bed. When a large bubble (one large enough to span the 1/2-inch ID tube from wall to wall) is formed with solids above it, the bubble will move up and down in an oscillatory fashion for long periods of time, allowing solids to leak past it. This does not upset the control. In larger diameter tubes, bubbles capable of spanning the tube probably will not exist.

It appears necessary for good control to remove air bubbles from the lines connecting the contactor and the differential pressure transducer. This was normally accomplished by extensive line bleeding after each draining of the contactor.

3.5 Rate Indication

A desirable by-product of the control system would be that it indicate and record the rate of solids flow through the bottom flow system. An approximate indication and record are provided by a strip-chart recorder which records the output pressure of the pressure-differential controller. (This is the pressure signal sent to the control valve.) This pressure is high which the valve is open and solids are flowing, and low when the valve is closed and no solids are flowing.

This indication is not perfect because of the on-off characteristic of the valve. Due to this, the control is oscillatory. If the pressure recorder is connected directly to the output of the differential pressure recorder, the wide pressure fluctuations will cover, with ink, a wide band on the chart. On the other hand, if the pressure recorder pen is damped and a capacitance tank placed in the line between it and the controller, the recorder gives a slightly oscillatory reading which can be approximately correlated with the solids flow rate.

In a larger system, in which the control valve will have a significant throttling range, this type of flow indicator-recorder should be useful for monitoring contactor operation and diagnosing upsets.

3.6 Solids Bypassing

In the solids-liquid contactor, the principal solids flow is down the downcomers. Some of the solids, however, work their way through the bed of steel balls on the plates, and rain down from plate to plate. Those solids which thereby bypass the control leg are removed from the bottom of the contactor through the solids bypass leg shown in Figure 3. The fluid at the top of this leg is at a slightly higher pressure than the fluid at the same level in the downcomer (because of the pressure drop of process fluid flowing upward through the solids on the lowest plate). By careful adjustment of the place of reinsertion, it should be possible to drain these solids into the fluidized bed in the constant inventory leg, at the point where this higher pressure is exactly balanced by part of the pressure drop in the fluidized bed. A simpler solution, used here, was to provide a bypass leg as shown in Figure 3, and occasionally draining these solids into the gas-lift line. By proper design of the bottom plate and proper selection of operating conditions it should be possible to reduce the rate of bypassing of solids to such a level that this valve need be opened only once or twice per day.

3.7 Bed Level

The level of the top of the dense-phase fluidized bed can be held at any of a wide range of locations between the two pressure taps. This level is changed by changing the setpoint of the differential pressure controller, which controls the solids outflow from the bottom flow system.

The "normal" setting is for about one-inch water column, which corresponds to an interface about one foot above the lower pressure tap. The system was run with settings up to twice those mentioned above, but it appears that at the higher settings the control system has limited natural ability to recover from upsets. With the controller setting at one inch water column, the bottom flow system has functioned perfectly over a seven-hour period without attention, during which period the other contactor parameters were changed about once per hour. With a setting of 2-1/2-inches water column, the bottom flow system required adjustment after each change of other contactor parameters.

APPENDIX C

SLURRY AIR LIFT TESTS

THIS PAGE
WAS INTENTIONALLY
LEFT BLANK

APPENDIX C -- SLURRY AIR LIFT TESTS

It is necessary to have a successful slurry air lift if the operation of a continuous contactor is to be successful. This presents no problem; under all circumstances the solids could be transported from the bottom of the column to a place above its top with a small amount of transport air and lift water.

To minimize the quantities of air and water transported from bottom to top of the column, small-diameter lift lines were used. A brief literature survey [3, 5, 6, 7, 8, and 9] indicates that very few lift studies have been conducted in pipe smaller than 1/2-inch ID. In this project a 1/4-inch IPS pipe was used as the lift line. Therefore, it is of interest to record its behavior.

1. PERFORMANCE OF 1/4-INCH PIPE AIR LIFT

The system for which these data are reported (shown in Figure C-1) consists of a 14-foot-long, vertical section of 1/4-inch IPS pipe; facilities for metering air and water into its base; a vapor-liquid separator at the top; and a pressure gauge at the bottom; a vapor-liquid separator at the top; and a pressure gauge at the bottom. No solids were used in the tests conducted in this unit.

Fig. C-1 One-fourth-inch pipe gas lift.

For various combinations of air and water flow rates the pressure at the bottom was recorded (Figure C-2). For each liquid flow rate, increasing the gas flow rate from zero to some high value results in a decrease in the bottom pressure down to some minimum, and then an increase, which seems to be unbounded. The same data are reinterpreted in terms of the "submergence" in Figure C-3. Here the term "submergence" is defined as the pressure at the bottom divided by the pressure of the system when full of static liquid. This is analogous to the submergence normally defined for gas lifts, but not exactly the same because the behavior of the mixing nozzle is not included.

2. COMPARISON WITH PUBLISHED CORRELATIONS

These results are not predicted by published correlations, which are based on data for larger pipe diameters. Chamberlain and Paige [5] correlated

their data for air lifts in 1/2-inch IPS to 2-inch IPS pipes by the equation

$$\left(\frac{V_a}{V_1} - 0.8\right) = 0.21 \left(\frac{D \times (1.6D + 2)}{S^{3/2} V_1}\right)^{0.94} \quad (8)$$

Here V_a is the air flow rate in ft^3/min , V_1 is the liquid flow rate in ft^3/min , D is the pipe diameter in feet, and S is the fraction of submergence, dimensionless. The data from Figure C-2 are replotted on appropriate co-ordinates in Figure C-4 and compared with Chamberlain and Paige's correlation. The agreement is very poor. Chamberlain and Paige did not claim that their correlation would work for pipe sizes outside the range they tested; Figure C-4 shows that it does not work for 1/4-inch IPS pipe.

Govier and his co-workers [3] correlated their data for pipe diameters between 0.63-inch ID and 2.50-inch ID by a plot of a modified friction factor

$$f' = \frac{g_c D}{2(U_1)^2} \left(1 + \frac{V_a}{V_1}\right) \left(\frac{-1}{\rho f} \frac{\Delta p}{\Delta h} - \frac{\frac{V_e}{V_a} + \frac{\rho g}{\rho f}}{\frac{V_e}{V_a} + 1}\right) \quad (9)$$

versus DU_1 with $D^{3.5}U_g$ as a parameter. Here g_c is the force-mass conversion factor, $32 \text{ ft lb}_m/\text{lb}_f \text{ sec}^2$; ρg is the gas density, in lb_m/ft^3 ; U_1 is the liquid superficial velocity, ft/sec ; and U_g is the gas superficial velocity, ft/sec . (Note that these symbols have been changed from those in the original article.) The data from Figure C-2 are replotted on these co-ordinates in Figure C-5 and compared with Govier et al's curve for $D^{3.5}U_g = 0.0001 \text{ ft}^{4.5}/\text{sec}$. Figure C-5 shows that there is rough agreement between the correlation of Govier et al and the 1/4-inch IPS data, but that the agreement is far from exact. The 1/4-inch pipe data do not seem to form lines of constant slope, as Govier's data do, but rather have a (minus) slope increase with decreasing gas flow rate. None of the lines shown has as steep a slope as the data shown by Govier et al. Note also that there is a vertical displacement of up to a factor of 10 between Govier's data for $D^{3.5}U_g = 10^{-4} \text{ (ft}^{4.5}/\text{sec)}$ and the same data measured in a 1/4-inch IPS pipe.

Fig. C-4 Comparison of 1/4-inch IPS gas lift with correlation of Chamberlain and Paige[5].

Fig. C-5 Comparison of 1/4-inch IPS gas lift performance with correlation of Govier et al[3].

($\text{ft}^{4.5}/\text{sec}$) and the same data measured in a 1/4-inch IPS pipe.

Chamberlain and Paige [5] indicate that for 1/2-inch IPS to 2-inch IPS pipe the air rate corresponding to the maximum liquid flow rate is given approximately by

$$V_a \text{ (scfm)} = 3 \text{ (Nominal pipe diameter, inches)}^2. \quad (10)$$

If nominal pipe diameter were the same as the actual diameter in inches (which is approximately true), this is equivalent to saying that the superficial air velocity at the maximum liquid flow rate is 9.2 ft/sec. However, for the 1/4-inch IPS tests, the superficial velocity corresponding to the maximum liquid flow rates is about 2.5 ft/sec. The bench-scale tests (described in the next section) also indicated that the maximum liquid rate occurred at a gas superficial velocity of 2 to 3 ft/sec.

In summary, the empirical correlations proposed in the literature for large diameter gas lift performance do not seem to be applicable to small diameter gas lifts.

3. BENCH-SCALE SLURRY GAS LIFTS

The effect of slurries on gas lift performance was tested in the bench-scale gas lift tester shown in Figure C-6. The slurry was formed by fluidizing solids in a tank with water. A small glassware lift was then used to determine the liquid flow rate for various slurry compositions and air flow rates.

Fig. C-6 Bench-scale gas lift tester.

Air, from a rotameter, entered the air line, flowed down to its junction with the lift line, and then lifted the fluid to the receiver. From the receiver the fluid drained to the tank. When a steady rate had been established, the stopcock in the drain line was closed, and the volume flow rate determined by timing the rise in level in the graduate cylinder. Submergence was controlled by varying the liquid level in the tank with a siphon.

Two such devices were used; they differed only in the diameter of the glass tubing: 4.1-mm ID and 7-mm ID.

The performance of the smaller diameter lift (4.1-mm ID) was quite erratic. In all gas lifts the flow was oscillatory; in this lift the flow oscillations were sufficiently strong to force air out of the bottom of the four-inch-long line below the air-water mixing point. Furthermore, the performance was strongly affected by the capacity of the air line. This was tested by inserting a surge vessel of about 200 cc into the air line. The resulting liquid flow rates were measurably different than those obtained without this surge vessel.

The 7-mm-ID lift did not show this erratic performance. Under very few circumstances were bubbles ejected from the line below the air-water mixing point. The performance of this 7-mm-ID lift for the air-water-system is shown in Figure C-7. These curves are typical of air lift curves; for a given submergence there is an air flow rate at which the liquid flow is maximum and the flow rate curve for any given submergence lies above the curve for lower submergences.

Fig. C-7 Performance of 7-mm-ID gas lift for air-water system.

Figures C-8, -9, and -10 show the effect of two kinds of solid particles on the gas lift performance of the 7-mm-ID lift. Figure C-8 shows the volume lifted, at 62 percent submergence, for various volume percent solids in the slurry when the solid was Dowex-50. Figure C-9 shows the analogous performance at 62 percent submergence when the solid was a typical alumina calcine from the pilot plant waste calciner. Figure C-10 shows the performance on Dowex-50 resin at 37 percent submergence.

The principal effect of substituting a slurry for a pure liquid on a gas lift is probably to increase the viscosity of the fluid being pumped. Most measurements of slurry viscosity [10] indicate that the ratio (slurry viscosity/pure fluid viscosity) is primarily a function of the volume percent solids in the slurry. If this is correct, then a plot of (volume of slurry pumped/volume of water pumped at the same air flow rate and submergence) versus volume percent solids in the slurry should give a unique curve for both resin and slurry, over the whole range of air rates and submergences. Figure C-11 shows such a plot; all the data can be reasonably well represented by a single curve.

Fig. C-8 Performance of 7-mm-ID gas lift with slurry of Dowex-50 resin in water. Submergence = 0.62.

Fig. C-9 Performance of 7-mm-ID gas lift with slurry of alumina calcine in water. Submergence = 0.62.

The scatter of the data is probably due to the imprecise methods used to measure the flow rates and the volume percent solids. Note that the points furthest from the curve (at lower left) are for 1 scfh, where the air rate measurement is least reliable. There is no apparent effect of solid particle density (resin 1.3 g/cc, calcine 2.4 g/cc), of particle size distribution (resin closely sized, calcine wide size range), of air rate (1 to 10 scfh), nor of submergence (0.37 to 0.62).

The slurry collecting in the graduated cylinder contained fewer volume percent solids than the slurry from which the solids were lifted. This indicates that some liquid is bypassing the solids in the lift tube or its entrance. This is

Fig. C-10 Performance of 7-mm-ID gas lift with slurry of Dowex-50 resin in water. Submergence = 0.37.

Fig. C-11 Air lift flow reduction due to solids.

plausible; for the solids to enter the tube they must be dragged upward by fluid flowing past them up into the lift-tube entrance.

The conclusions of the bench-scale gas lift study are:

- (1) Slurries with up to 60 volume percent solids can be successfully gas lifted.
- (2) Increasing the solids loading of the slurry decreases the fluid flow rate, all other factors being equal.
- (3) The effect of solids on fluid flow rate is primarily a function of volume percent solids in the slurry.

THIS PAGE
WAS INTENTIONALLY
LEFT BLANK

APPENDIX D

RECOMMENDATIONS FOR FUTURE WORK

THIS PAGE
WAS INTENTIONALLY
LEFT BLANK

APPENDIX D -- RECOMMENDATIONS FOR FUTURE WORK

1. VALVE DEVELOPMENT

The special valves developed for this project made it possible to control small flow rates of slurries without plugging. The 0.156-inch-orifice diameter valves did not plug when processing Dowex-50 slurries. They did plug on screened alumina calcine, but the 0.300-inch-orifice diameter valve, (Figure D-1) which was built to remedy this fault did not plug with screened alumina calcine.

Fig. D-1 Valve stems and seats after use.

(A) 0.156-inch-ID orifice seat and stem. Stem has silver solder point machined to a 44 degree cone. After 100 hours circulating Dowex-50 resin, the only sign of wear is the polish on the left side of the stem, presumably caused by slight misalignment of valve and seat.

(B) 0.300-inch-ID orifice seat and stem. Both seat and stem are stainless, stem machined to a 44-degree cone. After 10 hours circulating alumina calcine both seat and stem show significant pitting, presumably caused by crushing hard calcine particles between seat and stem.

(C) 0.156-inch-ID orifice seat and stem. Stem has silver solder point, hand shaped to form cone. After 100 hours circulating Dowex-50 resin the only sign of wear is the ring-shaped groove in the stem where it has made contact with the sharp-edged seat.

These valves were satisfactory to allow the development and demonstration of the solids transport and control system. However, the valves have several drawbacks:

- (1) They do not give a tight shutoff with solids in the system. This is particularly true of valves A and B in Figure D-1. These valves give a tight shutoff when no solids are present, but they can trap solids between the stem and seat and fail to close tightly.
- (2) They break the solids. Dowex-50 resin, in passing through both valves A and C (Figure D-1) in series, was reduced in size by about one-half percent by weight per pass. If the contactor were used for a once-through operation (eg, calcine leaching) this would be no problem. But for a re-circulating operation, (eg, ion exchange) the small number of fines generated per pass would accumulate and eventually cause problems.

- (3) They wear quickly when processing hard calcine particles, (valve B, Figure D-1).

Further valve development should proceed along the following lines:

- (1) The "line-contact" valves (valve C in Figure D-1) should be further tested, particularly with hard materials like calcine.

These valves probably cause less attrition and have less wear than the surface-contact valves (valves A and B in Figure D-1).

- (2) Valves should be tried in which one or both of the seating surfaces is a soft material, like rubber.
- (3) Other valve types to try are ball valves, iris valves, and grooved-needle valves.

2. LARGER DOWNCOMER FROM BOTTOM PLATE

As discussed in the text, the solids flow rate is limited by the bottom downcomer. A larger diameter bottom downcomer should be substituted for the 1/2-inch ID downcomer now used, and the column tested over a larger range of flow rates.

3. CALCINE OPERATION

The system was tested only briefly using alumina calcine as the solids being processed. Valve plugging caused this system to be abandoned. It would be of interest to see how the system functioned with high density (2.3 g/cc) particles. Another attempt should be made to run calcine in the system using control valves with larger diameter orifices (eg, 0.300 inch versus 0.156).

4. BYPASSING STUDY

As discussed in Appendix B, bypassing of solids through the bottom plate is a minor problem in this system. This bypassing can probably be eliminated by increasing the fluid velocity through the holes in the bottom plate (using fewer or smaller holes) and/or by using more balls on that plate. These methods should be tried. Also it appears that this bypassing is a function of pulser frequency, but the equipment used here did not permit measurement of the solids bypassing rate. This effect should be measured.

5. SMALL DIAMETER FEED FLOW FLUIDIZED BED

It would be advantageous to use a smaller diameter fluidized bed in the feed flow system. This would decrease the flow of fluidizing water. A one-inch ID bed with the dip tubes and drawoff line replaced with side taps should be tried. A side-tap drawoff line, with no vertical section between the fluidized bed and the control valve should be tried. It may give more reproducible flow behavior than the vertical drawoff line used here.

6. CLOSED FEED FLOW SYSTEM LINE

The feed flow system discharged through an air gap into the feed funnel of the contactor. A closed connection should be tested to see if column pulsations affect the feed flow system.

7. AIR FLUIDIZED FEED FLOW SYSTEM

For some operations, eg, calcine leaching, it would be advantageous to use air instead of water as the fluidizing agent in the feed flow system. The calcine would presumably be delivered dry, and unnecessary contact with water would be avoided.

This idea was tested briefly in a mock-up of the feed flow system shown in Figure 2. Air was introduced directly into the fluidizing water line. The fluidized bed formed was nonuniform, with large air bubbles. Furthermore, the packed bed in the inventory section prevented the solids in the top of the fluidizing section from fluidizing smoothly because the packed bed restrained the solids from moving upward. In spite of these difficulties, the system did work, delivering pulses of solids and gas whenever the valve was open. With some development work on the air inlet and the vessel shape, the uneven fluidization described above should be remedied, and satisfactory feed flow performance obtained.

8. PRESSURE FEED FLOW SYSTEM

The feed flow system should be tried under pressure. If it is successful, it could be located alongside the column it feeds, saving headroom.

9. ELIMINATION OF MECHANICAL PULSER

The mechanical pulser used in the laboratory column may not be satisfactory for remote operation because of its many moving parts. A more reliable pulser should be developed, probably a pneumatic one.

10. SLURRY GAS LIFTS

The preliminary tests of slurry gas lifts made here should be extended to longer and larger diameter pipes.

**PHILLIPS
PETROLEUM
COMPANY**

ATOMIC ENERGY DIVISION