AN OPTIMIZATION STUDY OF AN 
INTERMITTENT-FLOW MULTISTAGE 
FLUIDIZED ION EXCHANGE COLUMN 
WITH FLUID DIODE DOWNCOMERS

BY

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An optimization study of an intermittent-flow multistage fluidized ion exchange column was performed using a stochastic approximation method. A new type of downcomer, a fluid diode, was designed and employed to alleviate liquid bypassing through the downcomer. The well known ion exchange system, H\(^+\)/Na\(^+\) exchange on Dowex 50W resin, was used in this work.

The volumetric efficiency of the system was optimized with regard to certain column and diode parameters. A maximum volumetric efficiency of 71.8 hr\(^{-1}\) was obtained for the following conditions:

- average liquid flowrate = 3661 ml/min;
- resin flowrate = 56.1 gm/min;
- plate spacing 11.43 cm;
- lateral diode displacement = 0.794 cm.

Experiments have shown that a 78.2% increase in volumetric efficiency was achieved by use of the fluidic diode downcomers.
ACKNOWLEDGEMENT

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Special thanks are extended to Dr. F. Souhrada, whose help proved invaluable to the completion of this thesis.

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1. INTRODUCTION

In recent years there has been a considerable amount of development of fluidic systems and devices. The initial work was aimed at developing controllers and logical devices which could be reliable in environments too severe for electrical equipment. These devices generally consisted of no moving parts and relied on the interaction of the fluidic streams with the walls and each other to produce the desired response.

This research topic is concerned with the fluidic analogue of an electrical diode, a device which offers far more resistance to flow in one direction, than to flow in the reverse direction. Recently, Thompson (29) has utilized the principle of a fluid diode in designing a countercurrent liquid-liquid contactor.

In this study the fluid diode is to be applied as a downcomer in a sieve plate solids-fluids contacting device.

The principle of controlled cycling has been applied by Souhrada (26) in designing a multistage fluidized bed ion-exchange column. The major difficulty encountered in his work was the large amount of liquid bypassing the sieve plate through the downcomer. This succeeded in decreasing plate efficiencies due to improper contact with ion exchange resin.

As an extension of his work, it has been proposed to put the advantages of a fluid diode into practice in designing a suitable downcomer.
Consequently, the aims of the present work are threefold:  
1) the design and construction of fluidic diodes,  
2) the determination of the suitability of the diodes in alleviating liquid bypass through the downcomer,  
3) the optimization of various column and diode parameters with regard to the efficiency of mass transfer between the fluid and suspended solids.  
Because of its well know chemical equilibrium, the H\(^+\)/Na\(^+\) exchange on Dowex 50W resin was selected as the ion exchange system to be employed in this work.
2. LITERATURE REVIEW

2.1 FLUID DIODE

The fluid diode was invented by Tesla (28) in 1920. It consisted of a channel with many branching and returning loops which would behave like a diode. In more recent time, Linderoth (18) has described an intake nozzle for an intermittent ram-jet engine which would have properties of a fluid diode.

With the growing interest in fluidic logic and fluid amplification systems, more complex fluidic devices have been reported; these include counters, hot-gas throttles, position sensors, timers, and turbine speed controllers (34).

Ringleb (23) has investigated the flow patterns of an incompressible fluid in fluid diodes; specifically in single cusp-diffusers. He has suggested the general form of a mapping function to generate these shapes, and has applied the general theory of standing vortices to the computation of velocity distributions along the walls of various cusp cavities.

Thompson (29) has utilized the principle of a fluid diode to develop a new type of intermittent flow contactor. In his closed channel water table work, he was able to obtain a pressure drop ratio (hard to easy direction) of 16:1 at Reynolds numbers of $10^4$. Thompson has studied both single phase and two phase flow in the contactor and has obtained some mass transfer data using the system n-butanol/water to measure the aqueous phase transfer coefficient. Volumetric mass transfer coefficients obtained with this apparatus were higher than those reported in the literature for similar systems taken in packed beds and spray columns. Thompson has suggested several applications where the contactor can offer advantages over conventional devices:
1) in systems where emulsification is a problem due to low interfacial tension or small density differences between phases;
2) where simultaneous extraction and reaction are being carried out and heat must be added to or removed from the system;
3) in corrosive environments where internal moving parts are undesirable.

2.2 ION EXCHANGE

Until a few years ago, the basic method of using ion exchanges had not changed from about 1900. Practically all ion exchange plants have employed the fixed bed method of operation, in which both the exhaustion and regeneration cycles are carried out in a single vessel. This batch type method has limited the use of ion exchange in chemical processing applications. The variability of the product, the extra capacity required to handle continuous feeds in batch operations, and large quantities of resin required have had the effect of increasing plant costs, thus making ion exchange impractical in many situations.

Considerable research has been made in the field of ion exchange, with the majority of the progress centered toward improving ion exchange materials. Modern synthetic resins have higher capacity, exhibit better leakage characteristics, and are mechanically stronger than the natural zeolites and synthetic inorganic zeolites.

Recently, progress has been made in the refinement of the process and equipment design. The trend has been toward utilizing the advantage of continuous operation, namely, a constant supply of product of uniform quality at reduced investment in space, capital and labor. The main problem to be overcome in most instances is the small density difference between the resin and solution. Some methods employed to overcome this problem consist in solution downflow, mechanical conveying of resin through the contactor, prevention of resin fluidization in the columns, and use of multi-stage fluidized beds.
Limited success has been achieved with mixer-settler units. These contactors, developed by Hiester et al. (9) and Read (22), are similar in principle to the systems in use for liquid-liquid and liquid-solids extraction.

Higgins (12) and his co-workers have designed a column which operates on solution downflow principle. Upward resin movement in the column is accomplished by means of hydraulic impulses to the base of the resin bed. In operation, the resin and solution alternate in flowing countercurrently through the column. Satisfactory separations of similar cations (Na⁺ and Li⁺) have been carried out in this apparatus.

Several methods for the mechanical conveyance of the resin countercurrent to the solution have been attempted. Screw type assemblies have been tried but were found to cause excessive attrition of the resin and short circuiting of the two phases past each other. McCormack and Howard (19) enclosed the resin in a permeable casing and conveyed it like a continuous sausage string through a counterflowing solution. Selke and Muendel (25) carried out continuous ion exchange in a similar manner by use of a belt of phosphorylated cotton using a process developed by Guthrie (8). Mihara and Terasaki (20) utilized a wire mesh coated with resin and circulated this mesh through saturant and regenerant tanks.

Arehart et al. (1) have described two types of columns developed at Oak Ridge National Laboratory. A continuous ion exchange column has been developed which makes use of a hydraulic ram to prevent fluidization. This has the effects of remaining on stream with no lost time in moving the resin, and of having lower H.E.T.S. since co-current flow and fluidization are not permitted. Also described is a semicontinuous column which utilized solution down flow. Periodically the flow of solution is shut off and a timer turns on a pump to cause resin movement.
Hiester et al. (11) describe a unit developed at Stanford Research Institute. This column utilizes two special motorized resin valves adapted from Stanton (27) to dewater, inject (top), and withdraw (bottom), a metered resin slug.

Weiss, McNeill and Swinton (31) have adopted the ore dressing operation of jigging and have adapted multideck jigs for continuous exchange. The equipment has large capacity and is readily available from mine equipment manufacturers. Although contact efficiencies are lower, the authors feel that the ruggedness, flexibility, and ease of maintenance more than balance the low efficiency.

In a recent publication, Levendusky (17) described the Graver countercurrent ion exchange process. After an eight year research program, a flexible ion exchange process was developed. The system was designed so that the absorption, desorption, separation, and wash cycles are controlled independently. Typical applications for the Graver C.I. process consist of recovery of copper from spinning solution, treatment of waste water, and purification of radioactive materials.

Turner and Church (30) have made use of the principles of countercurrent gas absorption in developing a continuous ion exchanger which consisted of a sieve plate column containing 16 plates. Results indicated that the column was relatively easy to operate at steady state and that worthwhile separations were achieved. The authors claimed that the column was rather difficult to fill initially and that a different design of plate and downcomer would be desirable in practice.

A pulsed column of similar design to that described by Turner has been developed by Grimmett and Brown (7). Souhrada (26) has applied the principle of controlled cycling to a continuous fluidized bed apparatus. The intermittent flow of fluid through a multistage column provided for the easy start-up of the column, and the elimination of moving parts from
the apparatus. Problems were encountered in liquid bypassing through the downcomer causing decreased contact between the liquid and solids phases which resulted in lower efficiency. Suitable design of the downcomer should eliminate this situation.
3. THEORY

3.1 FLUID DIODE

The flow patterns that are produced in properly designed cusp cavities are shown in figures 3.1 and 3.2. When the flow is in the direction A to B (high pressure drop direction), the high velocity stream is attached to the wall and is diverted into the cavity causing flow reversal. Thus the stream follows a highly tortuous path in which high pressure drop is to be expected due to the turbulence generated.

The most important design consideration that affects the flow in this direction is the curvature of the channel wall C. Should the radius of curvature be too small, the stream may separate and short circuit to the cusp endpoint, causing less turbulence to be generated. When the fluid flows in the low pressure drop direction, the main stream follows a more direct route through the channel. With correct design, the stream separates from the wall at the cusp endpoint with the formation of a stable vortex in each cavity. Incorrect cusp shape may cause the vortex to break down and a turbulent wake to develop thus increasing the pressure drop.

Ringleb (23) has investigated the flow of an incompressible inviscid fluid over single cusp cavities and has suggested a general form of mapping function to generate these shapes. Wall profiles and flow patterns may be obtained by integration of:

\[
\frac{dF}{d\bar{z}} = \frac{\bar{p} \left( \bar{p} - \bar{p}_2 \right)}{\left( \bar{p} - \bar{p}_1 \right) \left( \bar{p} - \bar{p}_2 \right)}
\]  

(3.1)

Where \( \bar{p} \) is a complex mapping variable.
Fig. 3.1 Flow Pattern: High Pressure Drop Direction

Fig. 3.2 Flow Pattern: Low Pressure Drop Direction
The complex parameters \( \xi_1, \xi_2, \xi_3 \) can be adjusted to change the nature of the flow field. The wall profile of the cavity is obtained by plotting \( y \) versus \( x \) for real values of \( \xi \), where:

\[
x + iy = F(\zeta) \tag{3.2}
\]

The following expressions have been suggested by Ringleb (23) and Thompson (29) as mapping functions by which cusp cavities can be obtained:

\[
F(\xi) = \xi + \frac{\xi^2}{(\xi - \xi_i)} \tag{3.3}
\]

\[
F(\xi) = \xi + \xi_i \log (\xi - \xi_i) \tag{3.4}
\]

where \( \xi = \xi_i + i\eta \).

Increasing \( \xi_i \) has the effect of increasing the amplitude of the cusp; while decreasing \( \eta \), shortens the length of the cusp cavity.

Ringleb's analysis is only helpful in designing the channel for flow in the low pressure drop direction since the inviscid model breaks down when flow reversal occurs. Therefore, Ringleb's theory serves mainly to establish the kind of shape that will permit a stable vortex to be established.

Thompson discovered that the stream flowing in the high pressure drop direction would remain attached and be deflected into the cavity provided the radius of curvature was four inches or greater. Although these wide-open channels had poor behavior in the forward flow and either failed to produce a vortex or produced a weak vortex with thicker and more turbulent wake, Thompson has shown that the wall attachment of the stream resulted in high pressure drops making these channels ideal contactors.
3.2 ION EXCHANGE

The countercurrent ion exchange process consists of the removal of ions from a liquid feed stream that is contacting a stream of ion exchange resin moving in the opposite direction. The exchange reaction is dependent on the liquid film mass transfer coefficient, which is a function of the liquid concentration.

In the intermittent-flow multistage operation adopted in this work, the resin is fluidized by the upward flow of liquid through the column. Downward resin movement from plate to plate is achieved during the "no liquid flow" period of the operating cycle.

Turner and Church (30) and Moyle (21) have shown that the McCabe-Thiele diagram can be applied to such an ion exchange process. A mass balance performed across a section of the column and the bottom gives:

\[ Rq_o(y - y_B) = Lc_o(x - x_B) \]  

(3.5)

where

- \( L \) = liquid flowrate;
- \( R \) = resin flowrate;
- \( c_o \) = total ion concentration in the liquid;
- \( q_o \) = total ion concentration in the resin;
- \( y = q/q_o \) and \( x = c/c_o \).

Since the average liquid and solid flowrates over the cycle period are constant, and the total ion contents in the liquid and solid phases are also unaltered in the column, it can be said that equation 3.5 is that of a straight operating line. The number of theoretical stages (NTS) can be determined from this diagram by a stepping off procedure involving alternate use of the operating line and equilibrium curve.
Fig. 3.3 Equilibrium and Operating Line Curves for a Typical Ion Exchange System
The height equivalent to a theoretical stage (HETS) is then obtained by dividing the total exchange height by the number of theoretical stages.

For the system under consideration, namely, H⁺/Na⁺ exchange on Dowex 50W resin, it has been shown (6) that the equilibrium constant given by

\[ K = \frac{y(c_0 - c)}{c(1 - y)} \quad (3.6) \]

remains practically constant for a given total solution normality \( c_0 \). Therefore, the H⁺/Na⁺ equilibrium can be described by the following expression:

\[ y = \frac{K}{(K - 1) + 1/x} \quad (3.7) \]

Gilliland and Baddour (6) have determined equilibrium constants for salt solutions of varying normality:

<table>
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<tr>
<th>Solution Normality</th>
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<tr>
<td>0.01</td>
<td>1.58</td>
</tr>
<tr>
<td>0.1</td>
<td>1.50</td>
</tr>
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<td>1</td>
<td>1.42</td>
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A plot of \( \log(c_0) \) vs. \( K \) is shown in figure 3.4. This graph can be represented by the following analytical expression:

\[ K = \frac{-\log(c_0) + 17.75}{12.5} \quad (3.8) \]

Optimization experiments are to be carried out to ascertain the influence of the downcomer and operating variables on the combined throughput and efficiency of mass transfer. The objective function to be maximized is the volumetric efficiency \( \eta_V \), defined
Fig. 3.4 Graph of Salt Solution Normality Versus Equilibrium Constant for Hydrogen/Sodium Exchange on Dowex 50W Resin
as follows:

\[ \eta_v = \frac{Q_L + Q_V}{\text{HETS}.F} \] (3.9)

where \( Q \) = flowrate (L - liquid, R - resin);
\( \text{HETS} \) = height equivalent to a theoretical stage;
\( F \) = column cross sectional area.

The maximum value of the volumetric efficiency can be interpreted as the smallest volume of apparatus required for the necessary separation.

3.3 OPTIMIZATION PROCEDURE

Having defined the objective function, a suitable optimization method must be chosen. The stochastic approximation method was selected. It is a procedure for optimizing a multidimensional system where the measured variable is subject to random errors. Continual use of past measurements are utilized to estimate the approximate position of the goal.

This procedure was introduced by Robbins and Monro (24) who applied the method to finding the root of a single variable function measured in the presence noise.

Kiefer and Wolfowitz (16) later employed the same method to determine the maximum of a unimodal function obscured by noise. Blum (2) extended the procedure to the multidimensional case and Kesten (15) and others have proposed improvements. Wilde (32) gave general principles for the formulation of the stochastic approximation method. A modification described in Appendix I has been formulated by Fabian (5). Himmelblau (14) has described the implementation of this method by applying the procedure to a sample problem in his book.
4. APPARATUS AND EXPERIMENTAL PROCEDURES

4.1 DESIGN OF FLUIDIC DIODE

For use as an efficient downcomer in the fluidized bed column, the fluidic element should offer a high resistance to the upward flow of liquid and enable the resin easy passage downward. On the basis of this criterion, and Thompson's (29) discussion of the liquid flow on his contactor, three slightly different cusp shapes were selected for experimentation.

The first fluidic diode (Fig. 4.1) is almost identical to the one utilized by Thompson (29) for his liquid-liquid contactor. Since the radius of curvature at point C is four inches, flow attachment to the wall should occur thereby insuring high pressure drops in the direction A to B. Due to the slight intrusion of the cusp endpoint into the channel, pressure drops in the B to A direction are expected to be higher. Longitudinal displacement between the cusps is 1.5 inches with an amplitude of 7/16 inch.

Except for the absence of the intrusion of the cusp endpoint into the channel, the second cusp (Fig. 4.2) is identical to the first. Pressure drops in the "easy" direction should be lower than for shape 1.

Diode shape 3 (Fig. 4.3) had a one inch longitudinal displacement between cusps and an amplitude of 7/16 inch. Since the radius of curvature of the channel wall at C is less than four inches, short-circuiting across to the cusp endpoint should occur. Although this short-circuiting should decrease the pressure drop, the increased number of cusps per length of test section may counteract this voiding effect and produce pressure drops comparable to shapes 1 and 2.
Fig. 4.1 Diode Test Shape No. 1

Fig. 4.2 Diode Test Shape No. 2

Fig. 4.3 Diode Test Shape No. 3
4.2 APPARATUS FOR PRESSURE DROP MEASUREMENTS

The test section (Fig. 4.4) consisted of a series of cusps grouped in a staggered arrangement with total length of 12 inches. Twelve cusp cavities of type 3 and eight of types 1 and 2 could be accommodated in the test section. Lateral cusp displacements based on the narrowest parts of the channel were adjusted by means of changeable top and bottom supporting plates. Five inch diameter plexiglass disks, 1/2 inch in width, were used to fasten the test channel to the flow stabilization sections. These calming sections consisted of 2 foot long plexiglass tubes with 1 inch inside diameter. Pressure drop readings were obtained by use of a Gilmont differential micromanometer capable of measuring pressure difference of up to 2 inches of mercury with a sensitivity of 0.001 inch. The distance between pressure taps on the two bottom plates used was 9 1/16 and 9 3/32 inches (± 1/64 inch). Epoxy cement was used to fasten the cusp section to the supporting plates and prevent leaks.

Water was pumped from the rectangular storage tank (Fig. 4.5) into the test conduit by means of a 1/4 h.p. centrifugal pump running at 1725 r.p.m. with a flowrate of 6 gallons per minute at a head of 25 feet. The storage tank had a capacity of 120 Imperial gallons. Liquid flow was controlled by a 1/2 inch PVC globe valve at the entrance to the rotameter.

In general plexiglass and PVC were used in the construction of the apparatus.
Fig. 4.4a  Top View of Pressure Drop Test Section

Fig. 4.4b  Front View of Pressure Drop Test Section
Fig. 4.5 Apparatus for Pressure Drop Experiments
4.3 EXPERIMENTAL PROCEDURE FOR PRESSURE DROP MEASUREMENTS

A certain cusp shape and lateral displacement were selected and the test channel was fastened to the flow stabilization sections with epoxy cement. The epoxy was then allowed to cure overnight.

The system was initially filled with water pumped from the storage tank. The pump was then turned off and the micromanometer was zeroed by adjustment of the micrometer barrel until the needle point came in contact with mercury level in the manometer.

The pump was switched on once again and pressure drop measurements taken for liquid flowrates ranging from 750 to 5600 ml/min. Upon completion of these measurements, the water lines were exchanged to enable pressure drop readings to be obtained for opposite flow through the channel. Flow fluctuations induced by the cyclic nature of the centrifugal pump were reduced to about ± 70 ml/min. This was accomplished by use of a pinch clamp to increase the downstream pressure in the water return line to the tank.

The procedure was repeated for the three diode shapes and for lateral displacements of 1/4 and 1/2 inch (± 1/32 inch.)

4.4 APPARATUS FOR MASS TRANSFER EXPERIMENTS

The complete apparatus which was utilized in the mass transfer experiments is shown in Fig. 4.6. The 4 inch I.D. plexiglass column was 3 feet in length and could accommodate 6 sieve plates. The plates were held in position in the column by means of a 1/4 inch O.D. stainless steel rod (Fig. 4.7). Two inch high baffles were positioned on the sieve plates dividing them into two sections: 1) a fluidization section and 2) a downcomer section.
Fig. 4.6 Apparatus for Mass Transfer Experimentation
Fig. 4.7 Multi-stage Ion Exchange Column
Fig. 4.8 Sieve Plate - Fluid Diode Unit
Epoxy cement was used to fasten the 60 mesh stainless steel screen onto the fluidization section which occupied about 70.9% of the total plate area. Onto the remaining portion was fastened a 2 7/8 inch (± 1/16 inch) fluidic diode downcomer which was 5/8 inch thick and had 1/4 inch supporting side plates. Grooves cut in the side plates allowed for variations of lateral cusp displacements from 0 to 5/8 inch (± 1/32 inch). Plexiglass rods, 1/2 inch O.D., were used to vary the plate spacing in 1/2 inch (± 1/16 inch) increments from 3 1/2 to 5 inches.

The column was run in a cyclic operation. A cycle consisted of two periods:

1) solution flow through the column, during which time fluidization of the resin occurred resulting in the overflow of the resin into the downcomer section;

2) solution return to the storage tank, thus allowing for downward resin movement from plate to plate through the downcomer.

During the solution flow period, the liquid was passed through the column at a flowrate which remained invariant throughout all experimental runs. The cycle timing, reproducible to within 1% (± 0.1 sec), was varied by an adjustable-cam timer which operated an air-actuated solenoid valve. The flowrate through the column was monitored with a rotameter and was controlled by adjusting the liquid flow through the bypass line. Flow fluctuations of about ± 35 ml/min were induced by the pump. The liquid overflowed from the top of the column into a cylindrical trough 6 inches in diameter and 4 1/2 inches in height. The solution was then passed
through a cyclone to remove the entrained solids before being discarded. The entrained resin particles were collected in a 250 ml. plastic graduated cylinder.

A screw feeder assembly was used to feed the 24 to 35 mesh resin into the column. The rate of solids feed could be adjusted by manual control of the motor r.p.m. The resin was collected in a 2 liter plastic graduated cylinder positioned below the column. Resin flowrates, reproducible to ± 5%, were obtained by timing the volumetric flow of the resin into the cylinder.

An on-off temperature controller maintained the tank temperature at 22°C ± 2°C. A 15 foot length of 3/8 inch O.D. stainless steel pipe was used as a cooling coil.

4.5 EXPERIMENTAL PROCEDURE FOR MASS TRANSFER EXPERIMENTS

A certain diode shape was selected for testing. The diodes were attached to the sieve plates by two #6 stainless steel machine screws and the lateral cusp displacement adjusted to the distance indicated by the optimization method. Next, the resin collector was filled with distilled water and positioned below the column which was maintained full of solution at all times. The one inch PVC globe valve separating the resin collector from the column was opened, thus allowing the air trapped in the one inch line between the valve and the graduated cylinder to escape through the column to the atmosphere. The sieve plates were then put on the supporting rod with the appropriate plate spacers and inserted into the column. The liquid timing cycle was selected and the cam timer turned on.

The pump was activated and the bypass flow adjusted to give the appropriate rotameter reading. The screw feeder was switched
on and the motor controller set to the reading indicated by the optimization method. The column was run in this manner until steady state had been reached.

A timer was then turned on to measure the volumetric flowrate of the resin into the graduated cylinder. Four 200 ml. samples of the effluent liquid stream were taken from the discharge line at three minute intervals. The time period required for the flow of a certain volume of resin was noted and the globe valve before the resin collector was closed. The pump and screw feeder were turned off and four 2 to 3 ml. (± 0.05 ml.) samples of resin were taken from the top of the resin collector. The effluent and resin samples were then analyzed for H\(^+\) concentration as described below. Preparation of the titrant solutions used in determining H\(^+\) and Na\(^+\) concentrations is described in Appendix III.

### 4.6 DETERMINATION OF INITIAL HYDROGEN CONCENTRATION

The storage tank was filled by passing water at a rate of 1 gpm through a mixed resin deionizer to remove the anions and cations present. This procedure caused a slightly residual acidic content in the tank water.

Four 100 ml. (± 0.05 ml.) samples of tank water were titrated against a 0.01 N NaOH solution to an endpoint pH of 7.00± 0.01 using a Fischer Automatic Titrimeter. A Fischer combination glass/calomel reference electrode was used in these titrations. Volumes of sodium hydroxide added were determined to ± 0.05 ml. The initial hydrogen concentration was given by:

\[
N_{H^+} = \frac{N_{NaOH} \times V_{NaOH}}{V_{sample}} \tag{4.1}
\]
4.7 DETERMINATION OF INITIAL SODIUM CONCENTRATION

While the storage tank was being filled, a total of 1 1/2 to 2 pounds of reagent grade sodium chloride was added to the water. This was accomplished by periodic addition of saturated salt solution over the 2 hour filling period.

To a 50 ml. (± 0.05 ml.) sample of tank water was added 50 ml. of a 1 N KNO₃ buffer solution. This solution was titrated against a 0.02 N AgNO₃/0.48 KNO₃ solution. The equivalence point of the titration was determined from a graph of millivolts versus ml. of AgNO₃ added. A sample graph is shown in figure 4.9. Millivolt readings were obtained by means of an Orion digital pH/mv meter used with an Orion chloride specific ion electrode (Model 94-17) and double junction reference electrode (Model 94-02). Silver nitrate volumes were determined to ± 0.05 ml.

Four samples of tank water were analyzed for sodium content each time the tank was filled.

4.8 RESIN REGENERATION AND DETERMINATION OF RESIN CAPACITY

About 4 liters of resin were placed in the regeneration column and 2 liters of dilute HNO₃ (35 wt%) were passed through the column at a rate of 0.5 liter/hr. The resin was then washed with distilled water until the effluent was no longer acidic. This was tested by titrating 100 ml. of effluent with sodium hydroxide to the phenolphthalein endpoint. Washing was continued until no more than 0.05 ml. of a 0.1 N NaOH solution was required for the endpoint to be reached.

Four 2-3 ml. (± 0.05 ml.) samples of resin were placed in beakers to which were added 100 ml. (± 0.05 ml.) of a 0.05 N NaCl solution. These were allowed to sit under constant stirring for
Fig. 4.9 Sample Graph of Millivolts Versus Ml. of AgNO₃ Added
about one half hour; after which time, the samples were titrated against a 0.1 N NaOH solution to an endpoint pH of 7.00 ± 0.01 using the automatic titrimeter.

The resin capacity was given by:

\[
q_o = \frac{N_{NaOH} \times V_{NaOH} \times EWH^+}{Y \times V_{Resin}}
\]

where

- \(q_o\) = initial resin capacity (meq H+ /gm dry resin);
- \(N_{NaOH}\) = normality of NaOH solution;
- \(V_{Resin}\) = volume of resin sample (ml.);
- \(V_{NaOH}\) = volume of NaOH added (ml.);
- \(EWH^+\) = equivalent weight of H+ (1.00797);
- \(Y\) = ratio of dry weight to wet volume
  
  \(= 0.418 \pm 0.001 \text{ gm dry resin} \quad \text{ml wet resin}\).

4.9 DETERMINATION OF FINAL SODIUM CONCENTRATION AND RESIN CAPACITY

Four 100 ml. (± 0.05 ml.) samples of the effluent stream were titrated as described in section 4.7. The final sodium concentration was determined as follows:

\[
Na^+_f = Na^+_o - (H^+_f - H^+_o)
\]

Four 2-3 ml. (± 0.05 ml.) samples of spent resin were taken from the top of the resin collector. Procedures outlined in section 4.8 were followed to obtain the resin capacity.

4.10 EXPERIMENTAL PLAN

The independent variables in this analysis were:

- \(X_1 = \frac{T_f}{T_{nf}}\) — liquid timing cycle (sec. liquid flow) (sec. no flow)
- \(X_2 = MC\) — motor controller setting;
- \(X_3 = FS\) — plate spacing (cm.);
- \(X_4 = LD\) — lateral diode displacement (cm.)
The dependent variable was the volumetric efficiency as defined by equation 3.9.

The range of variables within which the optimization procedure was carried out were:

<table>
<thead>
<tr>
<th>$X_1$</th>
<th>$A^i$ (Lower Limit)</th>
<th>$B^i$ (Upper Limit)</th>
</tr>
</thead>
<tbody>
<tr>
<td>$X_2$</td>
<td>5.0</td>
<td>10.0</td>
</tr>
<tr>
<td>$X_3$</td>
<td>8.89</td>
<td>12.7</td>
</tr>
<tr>
<td>$X_4$</td>
<td>0.237</td>
<td>1.032</td>
</tr>
</tbody>
</table>

The physical significance of the liquid timing cycle and motor controller setting is shown in figures 4.10 and 4.11. An increase in the average volumetric liquid flowrate during a cycle was achieved by increasing the liquid timing cycle. Variation of resin flowrate was obtained by adjustment of the r.p.m. of the motor. This was accomplished by changing the motor controller setting.

Preliminary experiments were carried out with the following objectives in mind:

1) to determine the actual liquid flowrate through the column which results in good fluidization;

2) to test the performance of the column under actual operating conditions;

3) and finally, to determine an estimate of the experimental error.
Fig. 4.10  Graph of Average Liquid Flowrate Over a Cycle Period Versus Liquid Timing Cycle for a Liquid Flowrate through the Column of 5.204 liters/minute
Fig. 4.11 Graph of Resin Flowrate Versus Motor Controller Setting
5. RESULTS AND DISCUSSION

5.1 PRESSURE DROP MEASUREMENTS

Visual observations ascertained the existence of stable vortices when the water was flowing in the low pressure drop direction. Shape 3 (Fig. 4.3) exhibited less turbulence in the main stream than either of the other two cusp cavities.

Wall attachment by the water stream was observed for both test elements 1 and 2 when the flow was in the high pressure drop direction. Wall detachment in the third fluidic element did not occur until the liquid had entered the cusp and the void space occupied about one half of the cavity.

Pressure drop results, shown in detail in Appendix II, are summarized in figure 5.1. The Reynolds number and friction factor are based on the narrowest part of the test conduit.

When the flow was in the "easy" direction, the pressure drop for the channel followed quite closely the pipe flow correlation (solid line on Fig. 5.1) and exhibited the characteristic behaviour in the transition from laminar to turbulent flow. However, reverse flow through the test section showed the friction factor to increase with Reynolds number in a manner quite unlike pipe flow. It can be seen from Fig. 5.2, that the pressure drop ratios also increase with increasing Reynolds numbers.

The pressure drop measurements were reproducible to about ± 5%. Maximum pressure drop ratios (high/low directions) for the range explored occurred at the highest flowrates attained.
Fig. 5.1. Friction Factor Versus Reynolds Number
Fig. 5.2. Ratio of Pressure Drops (High/Low) Versus Reynolds Number
These were:

<table>
<thead>
<tr>
<th>Cusp Cavity</th>
<th>Re</th>
<th>$\frac{\Delta P_h}{\Delta P_f}$</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>9750</td>
<td>18.3</td>
</tr>
<tr>
<td>2</td>
<td>9750</td>
<td>18.4</td>
</tr>
<tr>
<td>3</td>
<td>9750</td>
<td>20.5</td>
</tr>
</tbody>
</table>

These compare favorably to the results reported by Thompson (29) for water flow through his contactor.

As expected, diode shapes 1 and 2 behaved almost identically. Surprisingly however, fluidic element 3 also had pressure drops comparable to shapes 1 and 2. This fact would seem to indicate that the increased number of cusp cavities per length of test section has counteracted the short-circuiting expected since the radius of curvature at point C (Fig. 4.3) is less than four inches.

In light of the pressure drop results, it was not possible to predict which fluidic diode would act as the best downcomer. Mass transfer experiments were required to establish the suitability of each fluidic element to prevent liquid bypassing through the downcomers in the ion exchange column.

5.2 Preliminary Experiments

Preliminary hydrodynamic experiments were carried out to test the performance of the system and to determine certain operating characteristics. It was found that a liquid flowrate of 5204 ml/min. during the "solution flow" period resulted in satisfactory column operation. Lower flowrates produced poor resin fluidization, while higher liquid throughputs caused excessive entrainment and were too large to be contained in the overflow trough. An overall cycle time of 20 seconds was found satisfactory with regard to the filling
and emptying of the overflow trough. A steady flow of solids into the column was achieved by feeding the solids as a slurry and by maintaining at least a one inch height of distilled water above the resin in the storage hopper.

The results obtained from the preliminary mass transfer experiments are shown in Tables 5.1 to 5.4. It can be seen that the concentration measurements were accurate to ± 5%. In the first two runs, air bubbles were detected below the plates. This tended to increase the resistance to upward liquid flow through the screen thus resulting in higher liquid bypass through the downcomer, and lower efficiencies. This problem was overcome by keeping the liquid feed line to the column full at all times. An estimate of the overall experimental error is the difference between the measured and calculated solids flowrate. Upon elimination of the air bubbles from the system, this difference was reduced to an acceptable ± 10% (Tables 5.3 and 5.4).

5.3 MASS TRANSFER EXPERIMENTS

After the preliminary experiments were performed, the stochastic optimization procedure was carried out as described in Appendix I.

The first optimization cycle was executed initially without the diodes in order to obtain a basis for later comparisons. Cycle 1 was then carried out for each diode shape individually.

In the non-diode runs, the top three plates were readily flooded. This situation was caused by the large amount of liquid bypassing the sieve plates through the downcomer which resulted in poor fluidization on the top plates and decreased contact efficiencies.
Table 5.1

MASS TRANSFER EXPERIMENT ON MULTISTAGE FLUIDIZED BED COLUMN

CUSP NO. 2
COLUMN DIAM. = 4.0 IN.
PLATE SPACING = 10.16 CM.
LATERAL DISPLACEMENT = 0.635 CM.

<table>
<thead>
<tr>
<th>RESIN CAP. (MEQ)</th>
<th>H/GM RESIN</th>
<th>NA (MEQ/L)</th>
<th>H CONCEN (MEQ/L)</th>
</tr>
</thead>
<tbody>
<tr>
<td>INITIAL</td>
<td>FINAL</td>
<td>INITIAL</td>
<td>FINAL</td>
</tr>
<tr>
<td>4.4911</td>
<td>3.5398</td>
<td>11.2666</td>
<td>.2187</td>
</tr>
<tr>
<td>4.4020</td>
<td>3.5643</td>
<td>11.4276</td>
<td>.2241</td>
</tr>
<tr>
<td>4.5609</td>
<td>3.5589</td>
<td>11.4678</td>
<td>.2296</td>
</tr>
<tr>
<td>4.5232</td>
<td>3.5147</td>
<td>11.6650</td>
<td>.2241</td>
</tr>
</tbody>
</table>

TOTALS 17.9773 14.5777 45.8311 .8965 37.6751
AVGS. 4.4943 3.6444 11.4578 .2241 9.4188
STD. DEV. .0678 .1982 .1655 .0045 .0899

LIQUID FLOW RATE = 2602 ML/MIN
SOLIDS FLOW RATE MEAS. = 22.30 G/MIN
SOLIDS FLOW RATE CALC. = 28.15 G/MIN
DIFF. IN MEAS. AND CALC. SOLIDS FLOW RATE = -20.8 P

X(IN) = 1.0  X(OUT) = 0.198  Y(IN) = 0.0  Y(OUT) = 0.189

NTS = .92  HETS = 66.17 CM.  PLATE EFF. = 15.35 P
VOLUME EFF. = 29.86 1/HR
Table 5.2

MASS TRANSFER EXPERIMENT ON MULTISTAGE FLUIDIZED BED COLUMN

Cusp No. 2  Run No. 2
Column Dia.= 4.0 IN.  No. of Plates= 6
Plate Spacing= 10.16 CM.  Baffle Height= 5.08 CM.
Lateral Displacement= 635 CM.  Liquid Cycle Ratio= 1.50

<table>
<thead>
<tr>
<th>Resin Cap. (MEQ/ H/GM. Resin)</th>
<th>Initial</th>
<th>Final</th>
<th>Na (MEQ/L) Initial</th>
<th>H Conc. (MEQ/L) Final</th>
</tr>
</thead>
<tbody>
<tr>
<td>4.4911</td>
<td>3.1408</td>
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<td>11.2666</td>
<td>7.8280</td>
</tr>
<tr>
<td>4.4020</td>
<td>3.0687</td>
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<td>11.4276</td>
<td>7.8499</td>
</tr>
<tr>
<td>4.5609</td>
<td>3.2728</td>
<td>11.4678</td>
<td>11.4678</td>
<td>7.8499</td>
</tr>
<tr>
<td>4.5232</td>
<td>3.1545</td>
<td>11.6690</td>
<td>11.6690</td>
<td>7.8062</td>
</tr>
<tr>
<td>Totals</td>
<td>17.9773</td>
<td>12.6368</td>
<td>45.8311</td>
<td>31.3340</td>
</tr>
<tr>
<td>Avgs.</td>
<td>4.4943</td>
<td>3.1592</td>
<td>11.4578</td>
<td>7.8335</td>
</tr>
<tr>
<td>Std. Dev.</td>
<td>0.0678</td>
<td>0.0846</td>
<td>0.1655</td>
<td>0.0209</td>
</tr>
</tbody>
</table>

Liquid Flowrate= 3123 ML/Min
Solids Flowrate Meas.= 21.16 G/Min
Solids Flowrate Calc.= 17.80 G/Min
Diff. in Meas. and Calc. Solids Flowrate= 18.9 P

x(In)= 1.0  x(Out)= 0.336  y(In)= 0.0  y(Out)= 0.297

Nts= 0.84  Hets= 72.37 CM.  Plate Eff.= 14.04 P

Volume Eff.= 32.37 1/HR
# Table 5.3

**MASS TRANSFER EXPERIMENT ON MULTISTAGE FLUIDIZED BED COLUMN**

**Cusp No. 2**
- **Column Dia.** = 4.0 IN.
- **Plate Spacing** = 10.16 CM.
- **Lateral Displacement** = .635 CM.

**Run No. 3**
- **No. of Plates** = 6
- **Baffle Height** = 5.08 CM.
- **Liquid Cycle Ratio** = 1.50

<table>
<thead>
<tr>
<th></th>
<th>Initial</th>
<th>Final</th>
<th>Initial</th>
<th>Na (Meq/L)</th>
<th>Initial</th>
<th>Final</th>
</tr>
</thead>
<tbody>
<tr>
<td>Resin Cap. (Meq H/Gm Resin)</td>
<td>4.4911</td>
<td>2.4808</td>
<td>17.6242</td>
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<tr>
<td></td>
<td>4.4120</td>
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<td>17.7450</td>
<td>.0984</td>
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<td></td>
</tr>
<tr>
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<td>4.5609</td>
<td>2.4684</td>
<td>17.7852</td>
<td>.0939</td>
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<td></td>
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<tr>
<td></td>
<td>4.5322</td>
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<td>17.6645</td>
<td>.0984</td>
<td>14.0286</td>
<td></td>
</tr>
<tr>
<td><strong>Totals</strong></td>
<td>17.9773</td>
<td>9.9673</td>
<td>70.8189</td>
<td>.4100</td>
<td>54.7798</td>
<td></td>
</tr>
<tr>
<td><strong>Averages</strong></td>
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<td>.1025</td>
<td>13.6950</td>
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<tr>
<td><strong>Std. Dev.</strong></td>
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<td>.0461</td>
<td>.0735</td>
<td>.0052</td>
<td>.2483</td>
<td></td>
</tr>
</tbody>
</table>

**Liquid Flowrate** = 3123 ML/MIN
**Solids Flowrate Meas.** = 21.21 G/MIN
**Solids Flowrate Calc.** = 21.20 G/MIN
**Diff. in Meas. and Calc. Solids Flowrate** = .1 P

**X(In)** = 1.0  **X(Out)** = .232  **Y(In)** = 0.0  **Y(Out)** = .446
**Nts** = 1.36  **Hets** = 44.82 CM.  **Plate Eff.** = 22.67 P

**Volume Eff.** = 52.41  1/HR
Table 5.4

MASS TRANSFER EXPERIMENT ON MULTISTAGE FLUIDIZED BED COLUMN

**Cusp No. 2**  
**Column Diameter = 4.0 in.**  
**Plate Spacing = 12.70 cm.**  
**Lateral Displacement = 0.635 cm.**  

**Run No. 4**  
**No. of Plates = 6**  
**Baffle Height = 5.08 cm.**  
**Liquid Cycle Ratio = 1.50**

<table>
<thead>
<tr>
<th>RESIN CAP. (MEQ)</th>
<th>H/GM RESIN</th>
<th>NA (MEQ/L)</th>
<th>H CONCN (MEQ/L)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Initial</td>
<td>Final</td>
<td>Initial</td>
<td>Final</td>
</tr>
<tr>
<td>4.4911</td>
<td>2.4610</td>
<td>17.6242</td>
<td>15.6434</td>
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<td>4.4020</td>
<td>2.4673</td>
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<td>2.4490</td>
<td>17.6645</td>
<td>15.8957</td>
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<tr>
<td><strong>TOTALS</strong></td>
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<td><strong>70.8189</strong></td>
<td><strong>63.0781</strong></td>
</tr>
<tr>
<td><strong>AVGS.</strong></td>
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<td><strong>17.7047</strong></td>
<td><strong>15.7695</strong></td>
</tr>
<tr>
<td><strong>STD. DVN.</strong></td>
<td><strong>.0678</strong></td>
<td><strong>.0735</strong></td>
<td><strong>.1030</strong></td>
</tr>
</tbody>
</table>

**Liquid Flow Rate** = 3123 ml/min  
**Solids Flow Rate Meas.** = 25.80 g/min  
**Solids Flow Rate Calc.** = 24.03 g/min  
**Diff. In Meas. and Calc. Solids Flow Rate** = 7.4 P  

\[ X(\text{IN}) = 1.0 \quad X(\text{OUT}) = 0.115 \quad Y(\text{IN}) = 0.0 \quad Y(\text{OUT}) = 0.453 \]

\[ NTS = 1.87 \quad HETS = 40.83 \text{ cm.} \quad \text{PLATE EFF.} = 31.11 \text{ P} \]

**Volume Eff.** = 57.65 1/hr
On the flooded plates, resin downflow during the "solution flow" period occurred only after the solids had reached a height of about 1 1/2 inches in the downcomer section. On the non-flooded plates, resin downflow was achieved only during the "no liquid flow" period.

In the experiments performed with the diodes, flooding occurred only when plate spacings less than 11.43 cm. were used. The flooding was caused by the proximity of the downcomer to the plate below. This resulted in the blockage of the downcomer outlet by the accumulated resin on the lower plate. The diodes succeeded in significantly reducing the liquid bypass through the downcomer. Downward resin flow occurred when a solids height of about 1/2 inch was achieved in the downcomer section. Vortex motion of the resin particles in the diode was induced by the upward liquid stream.

Concentration measurements and column operating variables were input into the computer program listed in Appendix VI. Results of individual runs are shown in Appendix IV and the summary of the optimization experiments in Appendix V.

The first run of cycle 1 was repeated for diodes 2 and 3. As can be seen from Tables 5.5 to 5.8, the volumetric efficiencies for these experiments were reproducible to within 3%. Differences between measured and calculated solids flow rate varied from 0.1% to 10.4% with the average variation being about 4%.

Table 5.9 shows the operating conditions which resulted in maximum volumetric efficiencies for each fluidic diode. Cycle 2 of the optimization procedure was performed solely with diode no. 2 since it had slightly better efficiencies than the other fluidic elements.
Table 5.5

MASS TRANSFER EXPERIMENT ON MULTISTAGE FLUIDIZED BED COLUMN

CUSP NO. 2
COLUMN DIAM.= 4.0 IN.
PLATE SPACING= 10.16 CM.
LATERAL DISPLACEMENT= .556 CM.

RUN NO. 1A
NO. OF PLATES= 6
BAFFLE HEIGHT= 5.08 CM.
LIQUID CYCLE RATIO= 1.00

<table>
<thead>
<tr>
<th>RESIN CAP. (MEQ H/GM RESIN)</th>
<th>NA (MEQ/L)</th>
<th>H CONCN (MEQ/L)</th>
</tr>
</thead>
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<tr>
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<td>4.5232</td>
<td>2.9541</td>
<td>25.9246</td>
</tr>
<tr>
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<td>17.9962</td>
<td>12.0098</td>
</tr>
<tr>
<td>AVGS.</td>
<td>4.4990</td>
<td>3.0025</td>
</tr>
<tr>
<td>STD. DVN.</td>
<td>.0380</td>
<td>.1295</td>
</tr>
</tbody>
</table>

LIQUID FLOW RATE= 2602 ML/MIN
SOLIDS FLOW RATE MEAS.= 35.20 G/MIN
SOLIDS FLOW RATE CALC.= 37.14 G/MIN
DIFF. IN MEAS. AND CALC. SOLIDS FLOW RATE= -5.2 P

X(IN)= 1.0   X(OUT)= .177   Y(IN)= 0.0   Y(OUT)= .333
NTS= 1.30   HETS= 47.06 CM.   PLATE EFF.= 21.59 P
VOLUME EFF.= 42.32 1/HR
Table 5.6

MASS TRANSFER EXPERIMENT ON MULTISTAGE FLUIDIZED BED COLUMN

CUSB NO. 2
COLUMN DIAM. = 4.0 IN.
PLATE SPACING = 10.16 CM.
LATERAL DISPLACEMENT = .556 CM.

RUN NO. 18
NO. OF PLATES = 6
BAFFLE HEIGHT = 5.08 CM.
LIQUID CYCLE RATIO = 1.00

<table>
<thead>
<tr>
<th>RESIN CAP. (MEQ/H/GM RESIN)</th>
<th>NA (MEQ/L)</th>
</tr>
</thead>
<tbody>
<tr>
<td>INITIAL FINAL</td>
<td>INITIAL FINAL</td>
</tr>
<tr>
<td>4.4911 2.6698</td>
<td>26.0247 .0219</td>
</tr>
<tr>
<td>4.4487 3.1315</td>
<td>25.8245 .0328</td>
</tr>
<tr>
<td>4.5331 2.9882</td>
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</tr>
<tr>
<td>4.5232 3.0967</td>
<td>25.9246 .0219</td>
</tr>
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TOTALS  17.9962  12.1862  103.7985 .1039  86.8963
AVGS.    4.4990  3.0466  25.9496 .0260  21.7241
STD. DVN. .0380  .0796  .0958  .0052  .1720

LIQUID FLOW RATE = 2602 ML/MIN
SOLIDS FLOW RATE MEAS. = 35.31 G/MIN
SOLIDS FLOW RATE CALC. = 38.87 G/MIN
DIFF. IN MEAS. AND CALC. SOLIDS FLOW RATE = -9.2 P

X(IN) = 1.0  X(OUT) = .164  Y(IN) = 0.0  Y(OUT) = .323
NTS = 1.33  HETS = 45.84 CM.  PLATE EFF. = 22.17 P
VOLUME EFF. = 43.52 1/HR
Table 5.7

MASS TRANSFER EXPERIMENT ON MULTISTAGE FLUIDIZED BED COLUMN

Cusp No. 3

**Column Diameter = 4.0 IN.**
**Number of Plates = 6**
**Plate Spacing = 10.16 CM.**
**Lateral Displacement = 0.556 CM.**

**Run No. 1A**
**Number of Plates = 6**
**Baffle Height = 5.08 CM.**
**Liquid Cycle Ratio = 1.00**

<table>
<thead>
<tr>
<th>Resin Cap. (MEQ H/GM Resin)</th>
<th>Initial</th>
<th>Final</th>
<th>Na (MEQ/L) Initial</th>
<th>H Conc'n (MEQ/L) Initial</th>
<th>Final</th>
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<td>Na (MEQ/L) Initial</td>
<td>H Conc'n (MEQ/L) Initial</td>
<td>Final</td>
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<tr>
<td>4.4928</td>
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<tr>
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<td>27.0056</td>
<td>0.0191</td>
<td>22.6051</td>
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<tr>
<td>4.5442</td>
<td>3.0009</td>
<td></td>
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<td>0.0246</td>
<td>22.6555</td>
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<tr>
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<td></td>
<td>27.1057</td>
<td>0.0219</td>
<td>22.6051</td>
</tr>
</tbody>
</table>

**TOTALS**
18.0019 11.7731 108.3628 .0875 90.4205

**AVGS.**
4.5005 2.9433 27.0907 .0219 22.6051

**STD. DVN.**
.0462 .0488 .0871 .0022 .0411

**Liquid Flowrate = 2602 ML/MIN**
**Solids Flowrate Meas. = 35.67 G/MIN**
**Solids Flowrate Calc. = 37.74 G/MIN**
**Diff. in Meas. and Calc. Solids Flowrate = -5.5 P**

**X(IN) = 1.0**  **X(OUT) = -166**  **Y(IN) = 0.0**  **Y(OUT) = 0.346**

**NTS = 1.38**  **HETS = 44.05 CM.**  **PLATE EFF. = 23.07 P**

**VOLUME EFF. = 45.24 1/HR**
Table 5.8

MASS TRANSFER EXPERIMENT ON MULTISTAGE FLUIDIZED BED COLUMN

CUSP NO. 3
COLUMN DIAM. = 4.0 IN.
PLATE SPACING = 10.16 CM.
LATERAL DISPLACEMENT = 0.556 CM.

RUN NO. 1B
NC.CF PLATES = 6
BAFFLE HEIGHT = 5.08 CM.
LIQUID CYCLE RATIO = 1.00

<table>
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<th>RESIN CAP. (MEQ/H/GM RESIN)</th>
<th>NA (MEQ/L)</th>
<th>H CONCN (MEQ/L)</th>
</tr>
</thead>
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<td>INITIAL</td>
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<tr>
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<tr>
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<td>2.9482</td>
</tr>
<tr>
<td>STD. DVN.</td>
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<td>.0696</td>
</tr>
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</table>

LIQUID FLOW RATE = 260.2 ML/MIN
SOLIDS FLOW RATE MEAS. = 35.64 G/MIN
SOLIDS FLOW RATE CALC. = 38.03 G/MIN
DIFF. IN MEAS. AND CALC. SOLIDS FLOW RATE = -6.3 P

X(IN) = 1.0   X(OUT) = .163   Y(IN) = 0.0   Y(OUT) = .345
NTS = 1.40   NETS = 43.59 CM.   PLATE EFF. = 23.31 P
VOLUME EFF. = 45.73 1/HR
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<th>$X_2$</th>
<th>$X_3$</th>
<th>$X_4$</th>
<th>$\eta_v$</th>
<th>$\frac{\eta_v - \eta_{vo}}{\eta_{vo}}$</th>
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</thead>
<tbody>
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<td>1</td>
<td>2.243</td>
<td>9.5</td>
<td>8.89</td>
<td>1.27</td>
<td>40.3</td>
<td>-</td>
</tr>
<tr>
<td>1</td>
<td>1</td>
<td>2.243</td>
<td>9.5</td>
<td>12.7</td>
<td>0.953</td>
<td>63.3</td>
<td>56.9%</td>
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<tr>
<td>2</td>
<td>1</td>
<td>2.243</td>
<td>9.5</td>
<td>12.7</td>
<td>0.953</td>
<td>66.8</td>
<td>65.6%</td>
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<tr>
<td></td>
<td>2</td>
<td>2.371</td>
<td>10.0</td>
<td>11.43</td>
<td>0.794</td>
<td>71.8</td>
<td>78.2%</td>
</tr>
<tr>
<td>3</td>
<td>1</td>
<td>2.243</td>
<td>9.5</td>
<td>12.7</td>
<td>0.953</td>
<td>62.4</td>
<td>54.8%</td>
</tr>
</tbody>
</table>
The maximum volumetric efficiency of 71.8 hr\(^{-1}\) was obtained using the second fluid diode with the following values of independent variables:

- Liquid timing cycle = 2.371
- Motor controller setting = 10
- Plate spacing = 11.43 cm.
- Lateral diode displacement = 0.794 cm.

This condition represented a 78.2% increase in volumetric efficiency over the non-diode experiments.

It should be noted that the upper bound on the solids flowrate was reached. Therefore, further experimentation would require that a method be devised to increase the resin flow into the column. This could possibly be accomplished by use of a wider pitch screw in the feeder system. However, having achieved the main goals of this research, namely, the design of a fluidic diode and the determination of its suitability in alleviating liquid bypass through the downcomer, it was felt that the additional experimentation required for further improvement in the optimum conditions was unjustified at this time.

Only five of the numerous physical and operating variables have been considered in this optimization study. Further experimentation along these lines should also include other variables such as diode length and width, baffle height, downcomer area, resin particle size, fluidization velocities, and column diameter. Predictive models incorporating these variables should be developed and tested.
6. CONCLUSIONS

An optimization study of an intermittent-flow fluidized ion exchange column was carried out. Liquid bypass through the downcomer sections of the six stage column was significantly reduced by use of fluid diode downcomers.

The manipulated variables consisted of the average liquid flowrate over a 20 second operating cycle, resin flowrate, plate spacing, lateral diode displacements, and diode shape. A maximum volumetric efficiency of 71.8 hr\(^{-1}\) was found for

- average liquid flowrate = 3661 ml/min,
- solids flowrate = 56 gm/min,
- plate spacing = 11.43 cm.,
- lateral diode displacement = 0.794 cm.,
- and fluid diode no. 2.

By use of the fluidic diode downcomers, a 78.2% increase in volumetric efficiency was achieved over the maximum efficiency obtained in the non-diode runs.
BIBLIOGRAPHY

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26. Souharada, F., unpublished work, Department of Chemical Engineering, University of New Brunswick, (1969)


28. Tesla, N., United States Patent 1,399,559 (1920)


34. Various Authors, Fluid Amplification Symposium, HDL (1964).
LIST OF SYMBOLS

\( c \) - concentration of \( Na^+ \) in bulk of solution (meq/l)

\( c_0 \) - solution normality (meq/l)

\( q_0 \) - initial resin capacity (meq/gm dry resin)

\( x \) - mole fraction of \( Na^+ \) in bulk of solution

\( y \) - mole fraction of \( Na^+ \) in resin

\( K \) - \( H^+/Na^+ \) equilibrium constant

\( L \) - liquid flowrate (ml/min)

\( R \) - solids flowrate (gm/min)

\( NTS \) - number of transfer stages

\( HETS \) - height equivalent to a transfer stage
APPENDIX I

The Stochastic Approximation Method
The Theory of the Method

A function $F$ is defined on set $A$, a part of Euclid space $\mathbb{R}(k)$. Points from set $A$ constitute the allowable conditions for the function $F$. $F$ is unknown, but for every allowable condition

$$x = [x^1, x^2, \ldots, x^k]$$

the value of $F(x) = F[x^1, x^2, \ldots, x^k]$ can be estimated. Let this measured or estimated value of $F$ be $y(x)$.

It should be noted that the function $F$ must be chosen carefully since, as in all optimization procedures, it should have a one local extremum.

Suppose that the set $A$ of allowable conditions is given by numbers $A^1, A^2, \ldots, A^k, B^1, B^2, \ldots, B^k$ so that if $x$ is a condition, then it is only allowable if

$$A^i \leq x^i \leq B^i$$

for $i = 1, 2, \ldots, k$ \hspace{1cm} (1)

During the experiment it will be necessary to change the condition by addition numbers to the various co-ordinates $x^i$.

Because of the closed interval $A^i$ and $B^i$, the following equations would be employed:

$$x^i + h^i = \begin{cases} x^i + h^i & \text{if } x^i + h^i \text{ lies within } (A^i, B^i) \\ A^i & \text{if } x^i + h^i < A^i \\ B^i & \text{if } x^i + h^i > B^i \end{cases}$$

(2)

Similar meaning is attached to $x^i - h^i$ since

$$x^i - h^i = x^i + (-h^i)$$

(3)
The search for the optimum condition $x$ (i.e. maximizing $F$) is carried out in cycles, each cycle consisting of steps. In the $n^{th}$ cycle the starting condition is $x_n$ and reaches $x_n + 1$ which is the starting condition for the $(n + 1)^{th}$ cycle. The $n^{th}$ cycle consists of steps $1, 2, \ldots s_n$ which are divided into testing and working steps. The former estimate the direction in which one should move in the $n^{th}$ cycle and the latter specify how far one should move to get to $x_n + 1$.

**The Choice of Starting Conditions**

Choose two positive numbers $c_i, a_i$ smaller than $\frac{1}{k} (B^i - A^i)$ for $i = 1, 2, \ldots k$ e.g.

$$c_i = \frac{1}{10} (B^i - A^i), \quad a_i = 2c_i$$

(4)

In the individual cycles the following values would be used

$$c_n^i = (-1)^{n+1} \frac{c_i}{n}$$

$$a_n^i = (-1)^{n+1} \frac{a_i}{n}$$

(5)

where $c_n^i$ is the length of the testing step and $a_n^i$ is the length of the working step. Finally it is necessary to choose $x_1$ for the starting condition. If there is no reason for a special choice, then

$$x_1^i = \frac{1}{k} (A^i + B^i)$$

(6)

**The Estimation of the Direction in the $n^{th}$ Cycle (testing steps)**

It is necessary to measure $y_n, 0; y_n, 1; y_n, 2; \ldots y_n, k$; where the conditions are defined as follows:
\[
\begin{align*}
    z_{x,0} &= x_n^{-1} - c_n^{-1}, x_n^2 - c_n^2, \ldots, x_n^{k-1} - c_n^{k-1}, x_n^{-k} - c_n^{-k} \\
    z_{x,1} &= x_n^{-1} + c_n^{-1}, x_n^2 - c_n^2, \ldots, x_n^{k-1} - c_n^{k-1}, x_n^{-k} - c_n^{-k} \\
    z_{x,2} &= x_n^{-1} - c_n^{-1}, x_n^2 + c_n^2, \ldots, x_n^{k-1} - c_n^{k-1}, x_n^{-k} - c_n^{-k} \\
    z_{x,k} &= x_n^{-1} - c_n^{-1}, x_n^2 - c_n^2, \ldots, x_n^{k-1} - c_n^{k-1}, x_n^{-k} + c_n^{-k}
\end{align*}
\]

For the values \(y_{n,i}\), the following values may be obtained

\[
\Delta_{n}^{i} = \begin{cases} 
+1 & \text{if } y_{n,i} - y_{n,0} > 0 \\
0 & \text{if } y_{n,i} - y_{n,0} = 0 \\
-1 & \text{if } y_{n,i} - y_{n,0} < 0
\end{cases}
\]

where \(\Delta_{n}^{i}\) is the sign of the partial derivative of the measured variable with respect to \(x_{n}^{i}\). Note that \(\Delta_{n}^{i} = +1\) or \(-1\) means that the direction of change for the \(i^{th}\) coordinate will be equal to the sign or opposite to the sign of \(a_{n}^{i}\).

**Working Steps**

Two modifications of the method may be used. They may be designated as \(\alpha_{0}\) and \(\alpha_{1}\).

For \(\alpha_{0}\)

\[
\begin{align*}
    x_{n+1} &= x_{n}^{i} + \Delta_{n}^{i} a_{n}^{i} \\
\end{align*}
\]

For \(\alpha_{1}\)

Let \(j = 1, 2, \ldots\), then

\[
\begin{align*}
    x_{n, k+j} &= x_{n}^{i} + j\Delta_{n}^{i} a_{n}^{i} \quad \text{for } i = 1, 2, \ldots, k \\
\end{align*}
\]

E.g.

\[
\begin{align*}
    x_{n, k+3} &= [x_{n}^{1} + 3\Delta_{n}^{1} a_{n}^{1}, \ldots, x_{n}^{k} + 3\Delta_{n}^{k} a_{n}^{k}]
\end{align*}
\]

The procedure then is to measure values of \(y_{n, j}\) for
j = k + 1, k + 2, … until the obtained values of \( y_n, j \) do not show a tendency to increase, that is,

\[
y_n, s_n - 1 > y_n, s_n, \text{ for } s_n > k + 1 \tag{11}
\]

in which case the progress in this \( n^{th} \) cycle is finished and

\[
x_{n+1} = x_n, s_n - 1 \tag{12}
\]

so that

\[
x_{i+1}^n = x_i^n + (s_n - 1) \Delta_i^1 a_i^1 \text{ for } i = 1, 2 \ldots k
\]

In the case where

\[
y_n^{i+2} > y_n^{i+1}, k+1
\]

in a number of successive cycles, the modification \( \varepsilon_0 \) may be employed.
APPENDIX II

Pressure Drop Results
Cusp No. ... 1 ...

Lateral Displacement = 0.635 cm.
Height of Cusp = 1.588 cm.
Pressure Tap Distance = 23.022 cm.

<table>
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<th>ML/Min</th>
<th>CM, of FWD</th>
<th>HG, REV</th>
<th>EQ. Dia CM</th>
<th>RE NO</th>
<th>F-Factor FWD</th>
<th>REV</th>
<th>REV/FWD</th>
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<td>0.0186</td>
<td>3.426</td>
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</table>
CUSP NO. ... 1 ...

LATERAL DISPLACEMENT = 1.270 CM.
HEIGHT OF CUSP = 1.588 CM.
PRESSURE TAP DISTANCE = 23.103 CM.

<table>
<thead>
<tr>
<th>ML/MIN</th>
<th>CH. OF FWRD</th>
<th>HG. REV</th>
<th>EQ. DIA CM.</th>
<th>RE NO</th>
<th>F-FACTOR FWRD</th>
<th>REV</th>
<th>REV/FWD</th>
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</thead>
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<td>0.0000</td>
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APPENDIX III

Preparation of Titrant Solutions
Preparation of Sodium Hydroxide Solution

A 0.20025 N (±0.00017 N) HCl standard was prepared by dilution of concentrated hydrochloric acid with distilled water and titrating against a 0.25 N (±0.1%) potassium hydroxide solution. The KOH standard was obtained by dilution, to 2 liters, of an ampoule containing 0.5 equivalents of KOH solution.

A 0.1 N NaOH solution was then prepared by dissolving approximately 16 gm. of reagent grade sodium chloride(97%) in 4 liters of distilled water. This was then titrated against the HCl standard to determine the NaOH solution normality.

A 0.01 N NaOH solution was prepared by dissolving about 1.6 gm. NaOH in 4 liters of distilled water and standardized against the HCl solution.

Preparation of Silver Nitrate Solution

The 0.02 N AgNO₃/0.48 N KNO₃ solution was prepared in the following manner.

About 7 gm. of silver nitrate and 100 gm. of potassium nitrate were dried separately at 110°C for two hours. A solution containing approximately 6.8 gm. AgNO₃ and 97.1 gm. KNO₃, (accurately weighed to ±0.001 gm.) dissolved in 2 liters of distilled water was made up. The AgNO₃ normality was then calculated from

\[
N_{\text{AgNO}_3} = \frac{W_{\text{AgNO}_3}/169.874}{2}
\]
APPENDIX IV

Results of Mass Transfer Experiments
# Mass Transfer Experiment on Multistage Fluidized Bed Column

**Cusp No. 0**
- **Column Diameter:** 4.0 IN.
- **Plate Spacing:** 10.16 CM.
- **Lateral Displacement:** 1.270 CM.

**Run No. 1**
- **No. of Plates:** 6
- **Baffle Height:** 5.08 CM.
- **Liquid Cycle Ratio:** 1.00

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**Liquid Flowrate:** 2602 ML/MIN

**Solids Flowrate Meas.:** 34.55 G/MIN

**Solids Flowrate Calc.:** 36.11 G/MIN

**Diff. in Meas. and Calc. Solids Flowrate:** -4.3 P

- **X(In):** 1.0
- **X(Out):** 0.375
- **Y(In):** 0.0
- **Y(Out):** 0.282
- **NTS:** 0.78
- **HETS:** 77.80 CM.
- **Plate Eff.:** 13.06 P
- **Volume Eff.:** 25.58 l/HR
MASS TRANSFER EXPERIMENT ON MULTISTAGE FLUIDIZED BED COLUMN

Cusp No. 0  Run No. 2
Column diam. = 4.0 in.  No. of plates = 6
Plate spacing = 10.16 cm.  Baffle height = 5.08 cm.
Lateral displacement = 1.270 cm.  Liquid cycle ratio = 1.50

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Liquid flowrate = 3123 ml/min
Solids flowrate meas. = 34.63 g/min
Solids flowrate calc. = 32.75 g/min
Diff. in meas. and calc. solids flowrate = 5.7 P

X(IN) = 1.0  X(OUT) = .401  Y(IN) = 0.0  Y(OUT) = .357
Nts = .82  Hets = 74.79 cm.  Plate eff. = 13.58 P
Volume eff. = 31.68 1/hr
MASS TRANSFER EXPERIMENT ON MULTISTAGE FLUIDIZED BED COLUMN

CUSP NO. 0
COLUMN DIAM.= 4.0 IN.
PLATE SPACING= 10.16 CM.
LATERAL DISPLACEMENT= 1.270 CM.

RUN NO. 3
NO. OF PLATES= 6
BAFFLE HEIGHT= 5.08 CM.
LIQUID CYCLE RATIO= 1.00

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LIQUID FLOWRATE= 2602 ML/MIN
SOLIDS FLOWRATE MEAS.= 43.84 G/MIN
SOLIDS FLOWRATE CALC.= 42.57 G/MIN
DIFF. IN MEAS. AND CALC. SOLIDS FLOWRATE= 3.0 P

X(IN)= 1.0   X(OUT)= .300   Y(IN)= 0.0   Y(OUT)= .267

NTS=.87   HETS= 70.47 CM.   PLATE EFF.= 14.42 P

VOLUME EFF.= 28.40 1/HR
MASS TRANSFER EXPERIMENT ON MULTISTAGE FLUIDIZED BED COLUMN

CUSP NO. 0
COLUMN DIAM. = 4.0 IN.
PLATE SPACING = 11.43 CM.
LATERAL DISPLACEMENT = 1.270 CM.

RUN NO. 4
NO. OF PLATES = 6
BAFFLE HEIGHT = 5.08 CM.
LIQUID CYCLE RATIO = 1.00

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LIQUID FLOW RATE = 2602 ML/MIN
SOLIDS FLOW RATE MEAS. = 35.15 G/MIN
SOLIDS FLOW RATE CALC. = 36.14 G/MIN
DIFF. IN MEAS. AND CALC. SOLIDS FLOW RATE = -2.8 P

X(IN) = 1.0  X(OUT) = .373  Y(IN) = 0.0  Y(OUT) = .282

HETS = 87.14 CM.  PLATE EFF. = 13.12 P
VOLUME EFF. = 22.83 1/HR
MASS TRANSFER EXPERIMENT ON MULTISTAGE FLUIDIZED BED COLUMN

CUSP NO. 0
COLUMN DIAM.= 4.0 IN.
PLATE SPACING= 10.16 CM.
LATERAL DISPLACEMENT= 1.270 CM.

RUN NO. 5
NO. OF PLATES= 6
BAFFLE HEIGHT= 5.08 CM.
LIQUID CYCLE RATIO= 1.00

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TOTALS                      17.9853 12.8271 112.4985 .0875 70.3348
AVGS.                       4.4963 3.2068 28.1246 .0219 17.5837
STD. DVN.                   .0195 .0104 .0233 .0045 .0637

LIQUID FLW RATE= 2602 ML/MIN
SOLIDS FLW RATE MEAS.= 35.05 G/MIN
SOLIDS FLW RATE CALC.= 35.44 G/MIN
DIFF. IN MEAS. AND CALC. SOLIDS FLW RATE= -1.1 P

X(IN)= 1.0         X(OUT)= .376   Y(IN)= 0.0     Y(OUT)= .287
NTS=.79          HETS= 77.45 CM.   PLATE EFF.= 13.12 P
VOLUME EFF.= 25.68 1/HR
MASS TRANSFER EXPERIMENT ON MULTISTAGE FLUIDIZED BED COLUMN

CUSP NO. 0
COLUMN DIAM.= 4.0 IN.
PLATE SPACING= 10.16 CM.
LATERAL DISPLACEMENT= 1.270 CM.

RUN NO. 6
NO. OF PLATES= 6
BAFFLE HEIGHT= 5.08 CM.
LIQUID CYCLE RATIO= 1.75

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LIQUID FLOW RATE= 3313 ML/MIN
SOLIDS FLOW RATE MEAS.= 48.63 G/MIN
SOLIDS FLOW RATE CALC.= 45.10 G/MIN
DIFF. IN MEAS. AND CALC. SOLIDS FLOW RATE= 7.8 F

X(IN)= 1.0 X(OUT)= .343 Y(IN)= 0.0 Y(OUT)= .302
NTS= .84 HETS= 72.51 CM. PLATE EFF.= 14.01 P
VOLUME EFF.= 34.92 1/HR
MASS TRANSFER EXPERIMENT ON MULTISTAGE FLUIDIZED BED COLUMN

CUSP NO. 0
COLUMN DIAM.= 4.0 IN.
PLATE SPACING= 8.89 CM.
LATERAL DISPLACEMENT= 1.270 CM.

RUN NO. 7
NO. OF PLATES= 6
BAFFLE HEIGHT= 5.08 CM.
LIQUID CYCLE RATIO= 2.24

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STD. DVN. .0195 .0594 .0233 .0045 .0413

LIQUID FLOW RATE = 3600 ML/MIN
SOLIDS FLOW RATE MEAS. = 56.20 G/MIN
SOLIDS FLOW RATE CALC. = 55.03 G/MIN
DIFF. IN MEAS. AND CALC. SOLIDS FLOW RATE = 2.1 P

X(IN) = 1.0  X(OUT) = .365  Y(IN) = 0.0  Y(OUT) = .260

NTS = .78  HETS = 68.47 CM.  PLATE EFF. = 12.98 P

VOLUME EFF. = 40.33 1/HR
MASS TRANSFER EXPERIMENT ON MULTISTAGE FLUIDIZED BED COLUMN

CUSP NO. 0
COLUMN DIAM.= 4.0 IN.
PLATE SPACING= 8.89 CM.
LATERAL DISPLACEMENT= 1.270 CM.

RUN NO. 8
NO. OF PLATES= 6
BAFFLE HEIGHT= 5.08 CM.
LIQUID CYCLE RATIO= 2.51

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LIQUID FLOW RATE= 3721 ML/MIN
SOLIDS FLOW RATE MEAS.= 58.83 G/MIN
SOLIDS FLOW RATE CALC.= 56.78 G/MIN
DIFF. IN MEAS. AND CALC. SOLIDS FLOW RATE= 3.6 P

X(IN)= 1.0  X(OUT)= .399  Y(IN)= 0.0  Y(OUT)= .246

NTS= .73  HETS= 73.31 CM.  PLATE EFF.= 12.13 P

VOLUME EFF.= 38.93 1/HR
MASS TRANSFER EXPERIMENT ON MULTISTAGE FLUIDIZED BED COLUMN

Cusp No. 1

Column Diameter = 4.0 IN.
Plate Spacing = 10.16 CM.
Lateral Displacement = .556 CM.

Run No. 1

No. of Plates = 6
Baffle Height = 5.08 CM.
Liquid Cycle Ratio = 1.00

Resin Cap. (MEQ H/GM Resin) | Na (MEQ/L) Initial | H Conc. (MEQ/L) Initial
---|---|---
Initial | Final | Initial | Final

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Totals = 17.9970 12.0534 108.3829 .0765 84.6651

Averages = 4.4993 3.0133 27.0957 .0191 21.1663

STD. Dev. = .0315 .0045 .0684 .0032 .0413

Liquid Flowrate = 2602 ML/MIN
Solids Flowrate Meas. = 35.01 G/MIN
Solids Flowrate Calc. = 37.03 G/MIN
Diff. in Meas. and Calc. Solids Flowrate = -5.5 P

X(IN) = 1.0  X(OUT) = .220  Y(IN) = 0.0  Y(OUT) = .330

Nts = 1.09  Hets = 55.68 CM.  Plate Eff. = 18.25 P

Volume Eff. = 35.77 1/HR
MASS TRANSFER EXPERIMENT ON MULTISTAGE FLUIDIZED BED COLUMN

CUSP NO. 1
COLUMN DIAM. = 4.0 IN.
PLATE SPACING = 10.16 CM.
LATERAL DISPLACEMENT = .556 CM.

RESIN CAP. (MEQ H/GM RESIN) INITIAL FINAL
4.4960 2.7094
4.4566 2.6883
4.5157 2.7574
4.5288 2.7269
TOTALS 17.9970 10.8820
AVGS. 4.4993 2.7205
STD. DYN. .0315 .0292

SOLIDS FLOWRATE MEAS. = 34.75 G/MIN
SOLIDS FLOWRATE CALC. = 37.64 G/MIN
DIFF. IN MEAS. AND CALC. SOLIDS FLOWRATE = -7.7 P

LIQUID FLOWRATE = 3123 ML/MIN

LIQUID CONCN (MEQ/L) INITIAL FINAL
27.0056 .0164 21.4195
27.1658 .0219 21.4701
27.0857 .0219 21.4701
27.1257 .0164 21.4701
TOTALS 108.3829 .0765 85.8297
AVGS. 27.0957 .0191 21.4574
STD. DYN. .0684 .0032 .0253

X(IN) = 1.0 X(OUT) = .209 Y(IN) = 0.0 Y(OUT) = .395
NTS = 1.33 MTS = 45.84 CM.
VOLUME EFF. = 51.87 1/HR

RUN NO. 2
NC. OF PLATES = 6
BAFFLE HEIGHT = 5.08 CM.
LIQUID CYCLE RATIO = 1.50

SOLIDS FLOWRATE CALC. = 37.64 G/MIN
DIFF. IN MEAS. AND CALC. SOLIDS FLOWRATE = -7.7 P

Y(IN) = 0.0 Y(OUT) = .395

PLATE EFF. = 22.17 P
MASS TRANSFER EXPERIMENT ON MULTISTAGE FLUIDIZED BED COLUMN

CUSP NO. 1
COLUMN DIAM.= 4.0 IN.
PLATE SPACING= 10.16 CM.
LATERAL DISPLACEMENT= .556 CM.

RUN NO. 3
NO. CF PLATES= 6
BAFFLE HEIGHT= 5.08 CM.
LIQUID CYCLE RATIO= 1.00

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LIQUID FLOW RATE = 2602 ML/MIN
SOLIDS FLOW RATE MEAS. = 41.82 G/MIN
SOLIDS FLOW RATE CALC. = 40.96 G/MIN
DIFF. IN MEAS. AND CALC. SOLIDS FLOW RATE = 2.1 P

X(IN) = 1.0  X(OUT) = .146  Y(IN) = 0.0  Y(OUT) = .327
NITS = 1.43  HETS = 42.55 CM.  PLATE EFF. = 23.88 P
VOLUME EFF. = 46.97 1/HR
MASS TRANSFER EXPERIMENT ON MULTISTAGE FLUIDIZED BED COLUMN

CUSP NO. 1
COLUMN DIAM. = 4.0 IN.
PLATE SPACING = 11.43 CM.
LATERAL DISPLACEMENT = .556 CM.

RUN NO. 4
NO. OF PLATES = 6
BAFFLE HEIGHT = 5.08 CM.
LIQUID CYCLE RATIO = 1.00

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<th>RESIN CAP. (MEQ/H/GM RESIN)</th>
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AVGS. 4.4993 2.8603 27.0937 .0191 22.4828
STD. DVN. .0315 .0477 .0684 .0032 .0413

LIQUID FLOW RATE = 2602 ML/MIN
SOLIDS FLOW RATE MEAS. = 34.43 G/MIN
SOLIDS FLOW RATE CALC. = 35.67 G/MIN
DIFF. IN MEAS. AND CALC. SOLIDS FLOW RATE = -3.5 P

X(IN) = 1.0  X(OUT) = .171  Y(IN) = 0.0  Y(OUT) = .364
NTS = 1.41  HETS = 48.58 CM.  PLATE EFF. = 23.53 P
VOLUME EFF. = 40.95 1/HR
MASS TRANSFER EXPERIMENT ON MULTISTAGE FLUIDIZED BED COLUMN

CUSP NO. 1
COLUMN DIAM. = 4.0 IN.
PLATE SPACING = 10.16 CM.
LATERAL DISPLACEMENT = .714 CM.

RUN NO. 5
NO. OF PLATES = 6
BAFFLE HEIGHT = 5.08 CM.
LIQUID CYCLE RATIO = 1.00

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TOTALS 17.9970 10.8078 117.4320 .0765 96.7167
AVGS. 4.4993 2.7019 29.3580 .0191 24.1792
STD. DEV. .0315 .0636 .1361 .0032 .0654

LIQUID FLOWRATE = 2602 ML/MIN
SOLIDS FLOWRATE MEAS. = 34.95 G/MIN
SOLIDS FLOWRATE CALC. = 34.98 G/MIN
DIFF. IN MEAS. AND CALC. SOLIDS FLOWRATE = -.1 P

X(IN) = 1.0  X(OUT) = .177  Y(IN) = 0.0  Y(OUT) = .399

NTS = 1.48  HETS = 41.32 CM.  PLATE EFF. = 24.59 P

VOLUME EFF. = 48.11 1/HR
MASS TRANSFER EXPERIMENT ON MULTISTAGE FLUIDIZED BED COLUMN

CUSP NO. 1
COLUMN DIAM. = 4.0 IN.
PLATE SPACING = 11.43 CM.
LATERAL DISPLACEMENT = .794 CM.

<table>
<thead>
<tr>
<th>RESIN CAP. (MEQ)</th>
<th>H/GM RESIN</th>
<th>NA (MEQ/L)</th>
<th>H CONCNR (MEQ/L)</th>
</tr>
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<tbody>
<tr>
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<td>INITIAL</td>
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<tr>
<td>4.5344</td>
<td>2.8711</td>
<td>29.5197</td>
<td>.0164</td>
</tr>
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</table>

TOTALS 18.0294 11.4183 117.4320 .0765 95.4001
AVGS. 4.5073 2.8546 29.3580 .0191 23.8500
STD. DVN. .0238 .0635 .1361 .0032 .0413

LIQUID FLOW RATE = 3313 ML/MIN
SOLIDS FLOW RATE MEAS. = 49.97 G/MIN
SOLIDS FLOW RATE CALC. = 47.77 G/MIN
DIFF. IN MEAS. AND CALC. SOLIDS FLOW RATE = 4.6 P

X(IN) = 1.0  X(OUT) = .188  Y(IN) = 0.0  Y(OUT) = .367
NTS = 1.34  HEFS = 51.12 CM.  PLATE EFF. = 22.36 P
VOLUME EFF. = 49.62 1/HR
### Mass Transfer Experiment on Multistage Fluidized Bed Column

**Cusp No. 1**
- **Column Diameter**: 4.0 in.
- **Plate Spacing**: 12.70 cm.
- **Lateral Displacement**: 0.953 cm.

**Run No. 7**
- **Number of Plates**: 6
- **Baffle Height**: 5.08 cm.
- **Liquid Cycle Ratio**: 2.24

#### Resin Cap. (meq H/gm Resin) Initial| Final | Na (meq/l) Initial| Final | H Conc. (meq/l) Initial| Final
---|---|---|---|---|---
4.5025| 2.8379| 26.3984| .0219| 26.1793
4.4775| 2.8014| 26.3175| .0164| 26.2300
4.5344| 2.8347| 26.5197| .0164| 26.2300
**TOTALS**| **18.0294**| **11.2729**| **117.4320**| **.0765**| **104.9199**
**AVGS.**| 4.5073| 2.8182| 26.3580| .0191| 26.2300
**STD. DEV.**| .0238| .0210| .1361| .0032| .0413

**Liquid Flowrate**: 3600 ml/min
**Solids Flowrate Meas.**: 58.61 g/min
**Solids Flowrate Calc.**: 55.86 g/min

**Diff. in Meas. and Calc. Solids Flowrate**: 4.9 P

**X(IN)** = 1.0  **X(OUT)** = 0.107  **Y(IN)** = 0.0  **Y(OUT)** = 0.375

**Nts** = 1.75  **Hets** = 43.66 cm.  **Plate Eff.** = 29.09 P

**Volume Eff.** = 63.28 1/HR
MASS TRANSFER EXPERIMENT ON MULTISTAGE FLUIDIZED BED COLUMN

CUSP NO. 1
COLUMN DIAM. = 4.0 IN.
PLATE SPACING = 12.70 CM.
LATERAL DISPLACEMENT = 1.111 CM.

<p>| | | | | |</p>
<table>
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<td>.1361</td>
<td>.0032</td>
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</table>

LIQUID FLOW RATE = 3721 ML/MIN
SOLIDS FLOW RATE MEAS. = 59.69 G/MIN
SOLIDS FLOW RATE CALC. = 60.81 G/MIN
DIFF. IN MEAS. AND CALC. SOLIDS FLOW RATE = -1.8 P

X(IN) = 1.0  X(OUT) = .213  Y(IN) = 0.0  Y(OUT) = .314

NTS = 1.07  HETS = 71.38 CM.  PLATE EFF. = 17.79 P

VOLUME EFF. = 40.09 1/HR
### MASS TRANSFER EXPERIMENT ON MULTISTAGE FLUIDIZED BED COLUMN

**Cusp No. 2**  
Column Diameter: 4.0 IN.  
Plate Spacing: 10.16 CM.  
Lateral Displacement: 0.556 CM.

**Run No. 1**  
No. of Plates: 6  
Baffle Height: 5.08 CM.  
Liquid Cycle Ratio: 1.00

<table>
<thead>
<tr>
<th>Resin Cap. (MEQ/H/GM Resin)</th>
<th>NA (MEQ/L)</th>
<th>H Conc. (MEQ/L)</th>
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<tr>
<td>4.5232</td>
<td>3.0241</td>
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</table>

**Totals**  
Liquid Flowrate: 2602 ML/Min

**Averages**  
Liquid Flowrate Calc.: 37.90 G/Min

**Standard Deviation**

<table>
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<tr>
<th>Resin Cap. (MEQ/H/GM Resin)</th>
<th>NA (MEQ/L)</th>
<th>H Conc. (MEQ/L)</th>
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**Liquid Flowrate**

**Solids Flowrate Meas.:** 35.26 G/Min  
**Solids Flowrate Calc.:** 37.90 G/Min  
**Diff. in Meas. and Calc. Solids Flowrate:** -7.0 P

**X(IN):** 1.0  
**X(OUT):** 0.170  
**Y(IN):** 0.0  
**Y(OUT):** 0.329

**NTS:** 1.31  
**HETS:** 46.37 CM.  
**Plate Eff.:** 21.91 P

**Volume Eff.:** 42.98 1/HR
# Mass Transfer Experiment on Multistage Fluidized Bed Column

## Cusp No. 2
- **Column Diameter:** 4.0 in.
- **Plate Spacing:** 10.16 cm.
- **Lateral Displacement:** 556 cm.

## Run No. 2
- **Number of Plates:** 6
- **Baffle Height:** 5.08 cm.
- **Liquid Cycle Ratio:** 1.50

## Statistical Data

<table>
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<tr>
<th>Resin Cap. (MEQ H/Gm Resin)</th>
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<td><strong>Totals</strong></td>
<td><strong>Averages</strong></td>
<td><strong>Stan. Dev.</strong></td>
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<td><strong>Averages</strong></td>
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<tr>
<td><strong>Stan. Dev.</strong></td>
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</tbody>
</table>

- **Liquid Flow Rate:** 3123 ml/min
- **Solids Flow Rate Meas.:** 35.89 g/min
- **Solids Flow Rate Calc.:** 35.39 g/min
- **Diff. in Meas. and Calc. Solids Flow Rate:** 1.4 P

- **X(IN):** 1.0  **X(OUT):** 0.163  **Y(IN):** 0.0  **Y(OUT):** 0.426
- **NTS:** 1.60  **HETS:** 38.12 cm.  **Plate Eff.:** 26.65 P
- **Volume Eff.:** 62.27 1/HR
# Mass Transfer Experiment on Multistage Fluidized Bed Column

**Cusp No. 2**
- **Column Diam.**: 4.0 in.
- **Plate Spacing**: 10.16 cm.
- **Lateral Displacement**: .556 cm.

**Run No. 3**
- **No. of Plates**: 6
- **Baffle Height**: 5.08 cm.
- **Liquid Cycle Ratio**: 1.00

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<thead>
<tr>
<th>Resin Cap. (MEQ)</th>
<th>H/GM Resin</th>
<th>NA (MEQ/L)</th>
<th>H ConcN (MEQ/L)</th>
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<td><strong>Totals</strong></td>
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</table>

**Liquid Flowrate**: 260.2 mL/min
**Solids Flowrate Meas.**: 45.54 g/min
**Solids Flowrate Calc.**: 46.77 g/min
**Diff. In Meas. and Calc. Solids Flowrate**: -2.6 P

<table>
<thead>
<tr>
<th>X(IN)</th>
<th>X(OUT)</th>
<th>Y(IN)</th>
<th>Y(OUT)</th>
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**NTS**: 1.45  **HETS**: 41.99 cm.  **Plate Eff.**: 24.20 P
**Volume Eff.**: 47.84 1/HR
MASS TRANSFER EXPERIMENT ON MULTISTAGE FLUIDIZED BED COLUMN

CUSP NO. 2
COLUMN DIAM.= 4.0 IN.
PLATE SPACING= 11.43 CM.
LATERAL DISPLACEMENT= .556 CM.

RUN NO. 4
NO. OF PLATES= 6
BAFFLE HEIGHT= 5.08 CM.
LIQUID CYCLE RATIO= 1.00

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<tr>
<th>RESIN CAP. (MEQ)</th>
<th>H/GM RESIN</th>
<th>NA (MEQ/L)</th>
<th>H CONCN (MEQ/L)</th>
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<td><strong>.0429</strong></td>
<td><strong>.0958</strong></td>
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</table>

LIQUID FLOW RATE = 2602 ML/MIN
SOLIDS FLOW RATE MEAS. = 35.89 G/MIN
SOLIDS FLOW RATE CALC. = 35.36 G/MIN
DIFF. IN MEAS. AND CALC. SOLIDS FLOW RATE = 1.5 P

X(IN) = 1.0  X(OUT) = .114  Y(IN) = 0.0  Y(OUT) = .376

NTS = 1.71  HETS = 40.10 CM.  PLATE EFF. = 28.50 P
VOLUME EFF. = 49.59 1/HR
MASS TRANSFER EXPERIMENT ON MULTISTAGE FLUIDIZED BED COLUMN

Cusp No. 2
Column Dia. = 4.0 in.
Plate Spacing = 10.16 cm.
Lateral Displacement = 0.714 cm.

Run No. 5
No. Of Plates = 6
Baffle Height = 5.08 cm.
Liquid Cycle Ratio = 1.00

<table>
<thead>
<tr>
<th>Resin Cap. (MEQ/Grn Resin)</th>
<th>NA (MEQ/L)</th>
<th>H Conc. (MEQ/L)</th>
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<tbody>
<tr>
<td>Initial</td>
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<td>Initial</td>
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<td>Averages</td>
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Std. Dev. | 0.0380 | 0.0480 | 0.0958 | 0.0052 | 0.0822 |

Liquid Flowrate = 2602 ML/Min
Solids Flowrate Meas. = 35.96 G/Min
Solids Flowrate Calc. = 37.12 G/Min

X(IN) = 1.0  X(OUT) = 0.136  Y(IN) = 0.0  Y(OUT) = 0.350

NTS = 1.54  HETS = 39.54 cm.  Plate Eff. = 25.70 P

Volume Eff. = 50.37 1/HR
MASS TRANSFER EXPERIMENT ON MULTISTAGE FLUIDIZED BED COLUMN

Cusp No. 2
Column Diameter = 4.0 in.
Plate Spacing = 11.43 cm.
Lateral Displacement = .794 cm.

Run No. 6
No. of Plates = 6
Baffle Height = 5.08 cm.
Liquid Cycle Ratio = 1.75

<table>
<thead>
<tr>
<th>Resin Cap. (MEQ H/GM Resin)</th>
<th>Na (MEQ/L)</th>
<th>H ConcN (MEQ/L)</th>
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Totals: 17.9962 12.0642 103.7985 .1039 92.4343

Averages: 4.4990 3.0160 25.9496 .0260 23.1086

Std. Dev.: .0380 .0504 .0052 .0411

Liquid Flowrate = 3313 ML/Min
Solids Flowrate Meas. = 50.15 G/Min
Solids Flowrate Calc. = 51.57 G/Min
Diff. in Meas. and Calc. Solids Flowrate = -2.7 P

X(IN) = 1.0  X(OUT) = .110  Y(IN) = 0.0  Y(OUT) = .330

Xts = 1.62  Hets = 42.22 cm.  Plate Eff. = 27.07 P

Volume Eff. = 60.25 1/HR
MASS TRANSFER EXPERIMENT ON MULTISTAGE FLUIDIZED BED COLUMN

CUSP NO. 2
COLUMN DIAM. = 4.0 IN.
PLATE SPACING = 12.70 CM.
LATERAL DISPLACEMENT = 0.953 CM.

RUN NO. 7
NO. OF PLATES = 6
BAFFLE HEIGHT = 5.08 CM.
LIQUID CYCLE RATIO = 2.24

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<th>INITIAL</th>
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LIQUID FLOWRATE = 3600 ML/MIN
SOLIDS FLOWRATE MEAS. = 58.92 G/MIN
SOLIDS FLOWRATE CALC. = 57.04 G/MIN
DIFF. IN MEAS. AND CALC. SOLIDS FLOWRATE = 3.3 P

X(IN) = 1.0   X(OUT) = .079   Y(IN) = 0.0   Y(OUT) = .350
NTS = 1.84   HETS = 41.40 CM.   PLATE EFF. = 30.68 P
VOLUME EFF. = 66.79 1/HR
HASS TRANSFER EXPERIMENT ON MULTISTAGE FLUIDIZED BED COLUMN

CUSP NO. 2

COLUMN DIAM.= 4.0 IN.

PLATE SPACING= 12.70 CM.

LATERAL DISPLACEMENT= 1.111 CM.

RESIN CAP. (MEQ)  H/GM RESIN  NA (MEQ/L)  H CONCNR (MEQ/L)
INITIAL FINAL INITIAL FINAL
4.4928 3.0870 27.0457 0.0219 23.0582
4.5389 2.9830 27.0056 0.0191 23.1086
4.5442 3.1030 27.2058 0.0246 23.2093
4.5259 3.1411 27.1057 0.0219 23.1086

TOTALS 18.0019 12.3141 108.3628 0.0875 92.4847
AVGS. 4.5005 3.0785 27.0907 0.0219 23.1212
STD. DVN. .0462 .0676 .0871 .0022 .0634

LIQUID FLOWRATE= 3721 ML/ MIN
SOLIDS FLOWRATE MEAS. = 65.24 G/MIN
SOLIDS FLOWRATE CALC. = 60.45 G/MIN
DIFF. IN MEAS. AND CALC. SOLIDS FLOWRATE= 7.9 P

X(IN)= 1.0  X(OUT)= .147  Y(IN)= 0.0  Y(OUT)= .316

NTS= 1.39  HETS= 54.66 CM.  PLATE EFF. = 23.23 P

VOLUME EFF. = 52.34 1/HR
MASS TRANSFER EXPERIMENT ON MULTISTAGE FLUIDIZED BED COLUMN

CUSP NO. 2
COLUMN DIAM. = 4.0 IN.
PLATE SPACING = 12.70 CM.
LATERAL DISPLACEMENT = 1.032 CM.

RUN NO. 9
NO. OF PLATES = 6
BAFFLE HEIGHT = 5.08 CM.
LIQUID CYCLE RATIO = 2.30

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TOTALS 17.8432 13.0950 79.3745 .0547 71.9659
AVGS. 4.4608 3.2737 19.8436 .0137 17.9915
STD. DVN. .0325 .0281 .1664 .0032 .0755

LIQUID FLOWRATE = 3626 ML/MIN
SOLIDS FLOWRATE MEAS. = 54.24 G/MIN
SOLIDS FLOWRATE CALC. = 54.92 G/MIN
DIFF. IN MEAS. AND CALC. SOLIDS FLOWRATE = -1.2 P

X(IN) = 1.0  X(OUT) = .094  Y(IN) = 0.0  Y(OUT) = .266
NTS = 1.56  HETS = 48.98 CM.  PLATE EFF. = 25.93 P
VOLUME EFF. = 56.77 1/HR
MASS TRANSFER EXPERIMENT ON MULTISTAGE FLUIDIZED BED COLUMN

CUSP NO. 2
COLUMN DIAM. = 4.0 IN.
PLATE SPACING = 12.70 CM.
LATERAL DISPLACEMENT = 1.032 CM.

RUN NO. 10
NO. OF PLATES = 6
BAFFLE HEIGHT = 5.08 CM.
LIQUID CYCLE RATIO = 2.17

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<td>Volume Eff.</td>
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# Mass Transfer Experiment on Multistage Fluidized Bed Column

**Cusp No. 2 Run No. 11**

- **Column Diameter:** 4.0 in.
- **Plate Spacing:** 12.70 cm.
- **Lateral Displacement:** 1.032 cm.
- **Number of Plates:** 6
- **Baffle Height:** 5.08 cm.
- **Liquid Cycle Ratio:** 2.30

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<tr>
<th>Resin Cap. (MEQ/H/GM Resin)</th>
<th>Resin H/GM Resin</th>
<th>N/A (MEQ/L) Initial</th>
<th>H Conc. (MEQ/L) Final</th>
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</table>

- **Totals:** 17.8432 13.0132 79.3745 .0547 69.2967
- **Averages:** 4.4608 3.2533 19.8436 .0137 17.3242
- **Stdev Dev.:** 0.0325 0.0829 0.1664 0.0032 0.0411

**Liquid Flowrate:** 3626 ML/MIN

**Solids Flowrate Meas.:** 52.29 G/MIN

**Solids Flowrate Calc.:** 51.98 G/MIN

**Diff. in Meas. and Calc. Solids Flowrate:** .6 P

**X(IN):** 1.0 **X(OUT):** .128 **Y(IN):** 0.0 **Y(OUT):** .271

**NTS:** 1.36 **HETS:** 55.96 CM. **Plate Eff.:** 22.69 P

**Volume Eff.:** 49.60 1/HR
MASS TRANSFER EXPERIMENT ON MULTISTAGE FLUIDIZED BED COLUMN

CUSP NO. 2
COLUMN DIAM. = 4.0 IN.
PLATE SPACING = 11.43 CM.
LATERAL DISPLACEMENT = 1.032 CM.

RUN NO. 12
NO. OF PLATES = 6
BAFFLE HEIGHT = 5.08 CM.
LIQUID CYCLE RATIO = 2.30

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<th>NA (MEQ/L)</th>
<th>H CONCN (MEQ/L)</th>
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<td>TOTALS 17.8432 13.3737</td>
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LIQUID FLOWRATE = 3626 ML/MIN
SOLIDS FLOWRATE MEAS. = 54.26 G/MIN
SOLIDS FLOWRATE CALC. = 58.87 G/MIN
DIFF. IN MEAS. AND CALC. SOLIDS FLOWRATE = -7.8 P

X(IN) = 1.0  X(OUT) = .086  Y(IN) = 0.0  Y(OUT) = .250
NTS = 1.57  HETS = 43.78 CM.  PLATE EFF. = 26.11 P
VOLUME EFF. = 63.68 1/HR
MASS TRANSFER EXPERIMENT ON MULTISTAGE FLUIDIZED BED COLUMN

CUSP NO. 2
COLUMN DIAM. = 4.0 IN.
PLATE SPACING = 12.70 CM.
LATERAL DISPLACEMENT = 0.873 CM.

RUN NO. 13
NO. OF PLATES = 6
BAFFLE HEIGHT = 5.08 CM.
LIQUID CYCLE RATIO = 2.30

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<tr>
<th>RESIN CAP. (MEQ H/GM RESIN)</th>
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TOTALS 17.8432 13.0438 79.3745 .0547 71.9659
AVGS. 4.4608 3.2609 19.8436 .0137 17.9915
STD. OVN. 0.0325 0.0142 0.1664 0.0032 0.0482

LIQUID FLOWRATE = 3626 ML/MIN
SOLIDS FLOWRATE MEAS. = 54.40 G/MIN
SOLIDS FLOWRATE CALC. = 54.33 G/MIN
DIFF. IN MEAS. AND CALC. SOLIDS FLOWRATE = .1 P

X(IN) = 1.0 X(OUT) = .094 Y(IN) = 0.0 Y(OUT) = .269
NTS = 1.56 HETS = 48.74 CM. PLATE EFF. = 26.06 P
VOLUME EFF. = 57.04 1/HR
**MASS TRANSFER EXPERIMENT ON MULTISTAGE FLUIDIZED BED COLUMN**

**CUSP NO. 2**  
**COLUMN DIAM. = 4.0 IN.**  
**PLATE SPACING = 11.43 CM.**  
**LATERAL DISPLACEMENT = .794 CM.**  

**RUN NO. 14**  
**NO. OF PLATES = 6**  
**BAFFLE HEIGHT = 5.08 CM.**  
**LIQUID CYCLE RATIO = 2.37**

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**LIQUID FLOWRATE = 3661 ML/MIN**  
**SOLIDS FLOWRATE MEAS. = 56.12 G/MIN**  
**SOLIDS FLOWRATE CALC. = 54.89 G/MIN**  
**DIFF. IN MEAS. AND CALC. SOLIDS FLOWRATE = 2.2 P**

**X(IN) = 1.0**  
**X(OUT) = .067**  
**Y(IN) = 0.0**  
**Y(OUT) = .277**

**NTS = 1.75**  
**HETS = 39.09 CM.**  
**PLATE EFF. = 29.24 P**

**VOLUME EFF. = 71.80 1/HR**
MASS TRANSFER EXPERIMENT ON MULTISTAGE FLUIDIZED BED COLUMN

Cusp No. 2
Column Dia. = 4.0 in.
Plate Spacing = 10.16 cm.
Lateral Displacement = 0.635 cm.

Run No. 15
No. of Plates = 6
Baffle Height = 5.08 cm.
Liquid Cycle Ratio = 2.51

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Liquid Flowrate = 3721 ml/min
Solids Flowrate Meas. = 56.37 g/min
Solids Flowrate Calc. = 55.88 g/min
Diff. in Meas. and Calc. Solids Flowrate = 0.9 P

X(IN) = 1.0  X(OUT) = 0.112  Y(IN) = 0.0  Y(OUT) = 0.263
NtS = 1.43  Hets = 42.64 cm.  Plate Eff. = 23.83 P
Volume Eff. = 66.91 1/HR
MASS TRANSFER EXPERIMENT ON MULTISTAGE FLUIDIZED BED COLUMN

CUSP NO. 3
COLUMN DIAM.= 4.0 IN.
PLATE SPACING= 10.16 CM.
LATERAL DISPLACEMENT= .556 CM.

RUN NO. 1
NO. OF PLATES= 6
BAFFLE HEIGHT= 5.08 CM.
LIQUID CYCLE RATIO= 1.00

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<th>RESIN CAP. (MEQ)</th>
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LIQUID FLOWRATE= 2602 ML/MIN
SOLIDS FLOWRATE MEAS.= 35.67 G/MIN
SOLIDS FLOWRATE CALC.= 37.86 G/MIN
DIFF. IN MEAS. AND CALC. SOLIDS FLOWRATE= -5.8 P

X(IN)= 1.0  X(OUT)= .165  Y(IN)= .0  Y(OUT)= .346
NTS= 1.39  HETS= 43.80 CM.  PLATE EFF.= 23.20 P
VOLUME EFF.= 45.50 1/HR
MASS TRANSFER EXPERIMENT ON MULTISTAGE FLUIDIZED BED COLUMN

CUSP NO. 3
COLUMN DIAM. = 4.0 IN.
PLATE SPACING = 10.16 CM.
LATERAL DISPLACEMENT = 0.556 CM.

RUN NO. 2
NO. OF PLATES = 6
BAFFLE HEIGHT = 5.08 CM.
LIQUID CYCLE RATIO = 1.50

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TOTALS 18.0019 10.5047 108.3628 0.0875 89.3633
AVGS. 4.5005 2.6262 27.0907 0.0219 22.3408
STD. DVN. 0.0462 0.0175 0.0871 0.0022 0.0252

LIQUID FLOWRATE = 3123 ML/MIN
SOLIDS FLOWRATE MEAS. = 35.22 G/MIN
SOLIDS FLOWRATE CALC. = 37.18 G/MIN
DIFF. IN MEAS. AND CALC. SOLIDS FLOWRATE = -5.3 P

X(IN) = 1.0  X(OUT) = 0.176  Y(IN) = 0.0  Y(OUT) = 0.416
NTS = 1.52  METS = 40.12 CM.  PLATE EFF. = 25.33 P
VOLUME EFF. = 59.25 1/HR
MASS TRANSFER EXPERIMENT ON MULTISTAGE FLUIDIZED BED COLUMN

Cusp No. 3
Column Diameter = 4.0 in.
Plate Spacing = 10.16 cm.
Lateral Displacement = 0.556 cm.

Run No. 3
No. of Plates = 6
Baffle Height = 5.08 cm.
Liquid Cycle Ratio = 1.00

<table>
<thead>
<tr>
<th>Resin Cap. (MEQ/H/GM Resin)</th>
<th>Na (MEQ/L)</th>
<th>H Conc. (MEQ/L)</th>
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Totals | 18.0019 | 0.0875 | 94.9516 |

AVGS. | 4.5005 | 3.0319 | 27.0907 | 0.0219 | 23.7379 |

STD. Dev. | 0.0462 | 0.0405 | 0.0871 | 0.0022 | 0.0291 |

Liquid Flowrate = 2502 ml/min
Solids Flowrate Meas. = 45.53 g/min
Solids Flowrate Calc. = 42.02 g/min
Diff. in Meas. and Calc. Solids Flowrate = 8.3 P

X(IN) = 1.0  X(OUT) = 0.125  Y(IN) = 0.0  Y(OUT) = 0.326

Nts = 1.54  Hets = 39.55 cm.  Plate Eff. = 25.69 P

Volume Eff. = 50.58 1/HR
MASS TRANSFER EXPERIMENT ON MULTISTAGE FLUIDIZED BED COLUMN

CUSP NO. 3
COLUMN DIAM.= 4.0 IN.
PLATE SPACING= 11.43 CM.
LATERAL DISPLACEMENT= .556 CM.

RUN NO. 4
NC.OF PLATES= 6
BAFFLE HEIGHT= 5.08 CM.
LIQUID CYCLE RATIO= 1.00

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TOTALS | 18.0019 | 10.7625 | 108.3628 | .0875 | 93.6426 |

AVGS. | 4.5005 | 2.6906 | 27.0907 | .0219 | 23.4107 |

STD. DYN. | .0462 | .0046 | .0871 | .0022 | .0411 |

LIQUID FLOWRATE= 2602 ML/MIN
SOLIDS FLOWRATE MEAS.= 34.46 G/MIN
SOLIDS FLOWRATE CALC.= 33.63 G/MIN
DIFF. IN MEAS. AND CALC. SOLIDS FLOWRATE= 2.5 P

X(IN)= 1.0  X(OUT)= .137  Y(IN)= 0.0  Y(OUT)= .402

NTS= 1.66  METS= 41.26 CM.  PLATE EFF.= 27.70 P

VOLUME EFF.= 48.12 1/HR
MASS TRANSFER EXPERIMENT ON MULTISTAGE FLUIDIZED BED COLUMN

Cusp No. 3  Run No. 5
Column Diam.= 4.0 IN.  No. of Plates= 6
Plate Spacing= 10.16 CM.  Baffle Height= 5.08 CM.
Lateral Displacement= .714 CM.  Liquid Cycle Ratio= 1.00

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Liquid Flowrate= 2602 ML/MIN
Solids Flowrate Meas.= 34.98 G/MIN
Solids Flowrate Calc.= 39.06 G/MIN
Diff. in Meas. and Calc. Solids Flowrate= -10.4 P

X(IN)= 1.0  X(OUT)= .149  Y(IN)= 0.0  Y(OUT)= .341
NTS= 1.45  HETS= 41.92 CM.  Plate Eff.= 24.24 P
Volume Eff.= 47.59 1/HR
MASS TRANSFER EXPERIMENT ON MULTISTAGE FLUIDIZED BED COLUMN

CUSP NO. 3
COLUMN DIAM.= 4.0 IN.
PLATE SPACING= 11.43 CM.
LATERAL DISPLACEMENT= .794 CM.

RUN NO. 6
NO. OF PLATES= 6
BAFFLE HEIGHT= 5.08 CM.
LIQUID CYCLE RATIO= 1.75

RESIN CAP. (MEQ H/GM RESIN) NA (MEQ/L) H CONCN (MEQ/L)
INITIAL FINAL INITIAL FINAL
4.4928 3.0484 27.0457 .0219 22.5548
4.4389 2.5556 27.0056 .0191 22.6555
4.5442 3.0352 27.2058 .0246 22.6051
4.5259 3.0032 27.1057 .0219 22.5548

TOTALS 18.0019 12.0424 108.3628 .0875 90.3702
AVGS. 4.5005 3.0106 27.0907 .0219 22.5925
STD. DVN. .0462 .0413 .0871 .0022 .0482

LIQUID FLOWRATE= 3313 ML/MIN
SOLIDS FLOWRATE MEAS.= 46.84 G/MIN
SOLIDS FLOWRATE CALC.= 50.19 G/MIN
DIFF. IN MEAS. AND CALC. SOLIDS FLOWRATE= -6.7 P

X(IN)= 1.0 X(OUT)= .167 Y(IN)= 0.0 Y(OUT)= .331

NTS= 1.34 HETS= 51.19 CM.

VOLUME EFF.= 49.64 1/HR
MASS TRANSFER EXPERIMENT ON MULTISTAGE FLUIDIZED BED COLUMN

CUSP NO. 3
COLUMN DIAM.= 4.0 IN.
PLATE SPACING= 12.70 CM.
LATERAL DISPLACEMENT= .953 CM.

RUN NO. 7
NO. OF PLATES= 6
BAFFLE HEIGHT= 5.08 CM.
LIQUID CYCLE RATIO= 2.24

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TOTALS: 17.9970 | 11.6586 | 108.3829 | .0765 | 97.4185
AVGS: 4.4993 | 2.9147 | 27.0957 | .0191 | 24.3546
STD. DVN: .0315 | .0327 | .0684 | .0032 | .0482

LIQUID FLOWRATE= 3600 ML/MIN
SOLIDS FLOWRATE MEAS.= 57.80 G/MIN
SOLIDS FLOWRATE CALC.= 55.28 G/MIN
DIFF. IN MEAS. AND CALC. SOLIDS FLOWRATE= 4.6 P

X(IN)= 1.0 X(OUT)= .102 Y(IN)= 0.0 Y(OUT)= .352
NTS= 1.72 HETS= 44.23 CM. PLATE EFF.= 28.71 P
VOLUME EFF.= 62.44 1/HR
MASS TRANSFER EXPERIMENT ON MULTISTAGE FLUIDIZED BED COLUMN

CUSP NO. 3
COLUMN DIAM. = 4.0 IN.
PLATE SPACING = 12.70 CM.
LATERAL DISPLACEMENT = 1.111 CM.

RUN NO. 8
NO. OF PLATES = 6
BAFFLE HEIGHT = 5.08 CM.
LIQUID CYCLE RATIO = 2.51

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TOTALS 17.9970 12.3180 108.3829 .0765 91.7798

AVGS. 4.4993 3.0795 27.0957 .0191 22.9450

STD. DEV. .0315 .0412 .0684 .0032 .0252

LIQUID FLOWRATE = 3721 ML/MIN

SOLIDS FLOWRATE MEAS. = 60.01 G/MIN

SOLIDS FLOWRATE CALC. = 60.09 G/MIN

DIFF. IN MEAS. AND CALC. SOLIDS FLOWRATE = -.1 P

X(IN) = 1.0 X(OUT) = .154 Y(IN) = 0.0 Y(OUT) = .316

XTS = 1.36 MTS = 56.06 CM. PLATE EFF. = 22.65 P

VOLUME EFF. = 51.02 1/HR
APPENDIX V

Summary of Optimization Experiments
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APPENDIX VI

Program Listings
PROGRAM TST (INPUT, OUTPUT, TAPE5=INPUT, TAPE6=OUTPUT)
COMMON /LAB1/ X(10,4), Y(10), D(4), A(4), DEL(4)

READ NUMBER OF DIODES
READ(5,1000) NOCSP
DO 5555 ICSP=1, NOCSP
READ NUMBER OF OPTIMIZATION CYCLES
READ(5,1000) NOCYC
DO 6666 ICYC=1, NOCYC

DATA INITIALIZATION
NCK=0
CALL DINIT(ICYC)
DO 9000 NFT=2,10

READ VALUES OF INDEPENDENT VARIABLES
READ(5,1100) NCUSP, (X(NPT,J), J=1,4)

MPTM2=NPT-1
IF(NCUSP.GT.3) GO TO 99

CALL VEFF(Y(NPT), NCUSP)
IF(NPT.EQ.2) GO TO 9000

CALL DELTA(NPT)
IF(NPT.LE.6) CALL DELTA(NPT)
IF(Y(NPT-1)-Y(NPT)) .GT. 9000,9000,36

9000 CONTINUE

CALCULATE VALUES OF INDEPENDENT VARIABLES FOR NEXT RUN

99 NCK=1
IF(NPT.LE.6) GO TO 999
NPTM2=NPT-2
IF(NPT.EQ.7) GO TO 30
IF(Y(NPT-2)-Y(NPT-1)) .LE. 30,30,35
30 DO 40 I=1,4
40 X(NPT,I)=X(1,I)+FLOAT(NPT-6)*DEL(I)*A(I)
GO TO 111
999 DO 50 I=1,4
50 X(NPT,I)=X(1,I)-D(I)
X(NPT,NPTM2)=X(1,NPTM2)+D(NPTM2)
111 CALL OUTPT(ICSP, ICYC, NPT)
GO TO 6666
35 CALL OUTPT(ICSP, ICYC,(NPT-1))
GO TO 37
36 CALL OUTPT(ICSP, ICYC, NPT)
READ(5,1000) NCUSP
37 DO 45 I=1,4
45 X(1,I)=X(NPTM2,I)
Y(I)=Y(NPTM2)
CALL DINIT(ICYC+1)
IF(NCK.EQ.0) GO TO 6666
CALL OUTPT(ICSP, (ICYC+1),2)
6666 CONTINUE
5555 CONTINUE
WRITE(6,2222)
2222 FORMAT(*1*////**1*)
STOP
END
SUBROUTINE DINIT(NN)
COMMON /LAB1/ X(10,4), Y(10), D(4), A(4), DEL(4)
DIMENSION U(4), E(4)
IF (NN.GT.1) GO TO 100
Y(1)=0.0
DO 5 I=1,4
     READ UPPER AND LOWER BOUNDS ON INDEPENDENT VARIABLES
1000 READ (5,1000) U(I), B(I)
     D(I)=0.1*(U(I)-B(I))
     X(1,I)=0.5*(U(I)+B(I))
     IF (I.LT.3) GO TO 5
     X(1,I)=X(1,I)*2.54
     D(I)=D(I)*2.54
     D(3)=0.25*2.54
     A(I)=2.0*D(I)
     GO TO 50
5   A(I)=2.0*D(I)
50  DO 20 I=1,4
     DEL(I)=0.0
20  X(2,I)=X(1,I)-D(I)
     DO 40 I=2,10
     Y(I)=0.0
     RETURN
    END

SUBROUTINE STAT(X,N,XBAR,XSUM,STDVN)
DIMENSION X(4)
XSUM=0.0
SSQ=0.0
DO 10 I=1,N
     SSQ=SSQ+X(I)*X(I)
10  XSUM=XSUM+X(I)
XBAR=XSUM/FLOAT(N)
STDVN=SQRT((SSQ-XBAR*XBAR*FLOAT(N))/FLOAT(N-1))
RETURN
END
SUBROUTINE VETAV(ETAV, NCUSP)
DIMENSION X(10), Y(10)
READ OPERATING PARAMETERS
READ(5,11) NE, NP, PL, H, F, D, T, QL, QS
FORMAT(2I5,7F10.3)
QL=QL*(T/(T+1))
PL=PL*2.54
H=H*2.54
D=D*2.54
WRITE(6,9) NCUSP, NE, NP, PL, H, D, T
Q=QL
WRITE(6,15) QL, QS, S, DELTAS, XN, Y1, PTS, HTS, ETAP, ETAV
READ CONC., DATA AND CALCULATION OF ION FRACTIONS
CALL DATARD(XOD, XND, C, Y1D, EK)
HTS=0
XO=1,
XL=XND/XOD
Y1=(C-Y1D)/C
A=Y1/(1.-XN)
Q=A*XN
J=0
Y(1)=Y1
CALCULATION OF NO. OF THEORETICAL STAGES IN COLUMN
DO 3 I=1, 10
X(I)=Y(I)/(EK-(EK-1.0)*Y(I))
IF(X(I)-XN)<5.5
4 Y(I+1)=A*X(I)-Q
J=J+1
5 IF(J)7, 7, 6
P=NP
6 PTS=J+((XN-X(I-1))/(X(I)-X(I-1)))
GO TO 8
7 PTS=(XN-XC)/(X(1)-XO)
CALCULATION OF SOLIDS FLOW RATE
S=QL*(XOD-XN))/(C-Y1D)
DELTAS=(QS-S)*100./S
P=NP
CALCULATION OF HETS, PLATE AND VOLUMETRIC EFFICIENCIES
HTS=(P/PTS)*PL
ETAP=PTS*100./P
ETAV=60.*((QL+S/0.418333)/(HTS*81.07))
WRITE(6,15) QL, QS, S, DELTAS, XN, Y1, PTS, HTS, ETAP, ETAV
1X(IN)=1.0, 4X, * X(OUT)=*, F6.3, 8X, * Y(IN)=-0.0, 4X,
RETURN
END
SUBROUTINE DATARO(XOD,XND,C,Y1D,EK)
DIMENSION RI(4),RF(4),SI(4),HI(4),HF(4)
EQCONS(CONC)=(-ALOG10(CONC)+17.75)/12.5
READ TITRANT SOLUTION NORMALITIES
READ(5,1000) SINAOH, SFNAOH, RINAOH, RFNAOH, SAGN03
WRITE(6,7000)
7000 FORMAT(/21X,*RESIN CAP. (MEQ H/GM RESIN) NA (MEQ/L) - H CONCN (M
1EQ/L) */19X,* INITIAL FINAL INITIAL FINAL INITIAL
DO 10 I=1,4
READ SOLUTION AND TITRANT VOLUMES, WEIGHT OF RESIN SAMPLES
READ(5,1100) W1, V1, W2, V2, W3, V3, W4, V4, W5, V5
1100 FORMAT(10F8.5)
RI(I)=RINAOH*V1/W1*1.00797
SI(I)=SAGN03*V2/W2*1.000,0
HI(I)=SINAOH*V3/W3*1.000,0
HF(I)=SFNAOH*V4/W4*1.000,0
RF(I)=RFNAOH*V5/W5*1.00797
WRITE(5,2000) RI(I), RF(I), SI(I), HI(I), HF(I)
5000 FORMAT(25X, F9.4, F10.4, F14.4, 2F11.4)
CALL STAT TO CALCULATE AVGS. AND STD. DEVNS. CF CONC. MEASUREMENTS
CALL STAT(RI,4, RIAVG, RISUM, RISTD)
CALL STAT(RF,4, RFAVG, RFSUM, RFSTD)
CALL STAT(SI,4, SIAVG, SISUM, SISTD)
CALL STAT(HI,4, HIAVG, HISUM, HISTD)
CALL STAT(HF,4, HF AVG, HFSUM, HFSTD)
PRINT CONCENTRATION RESULTS
WRITE(6,5200) RISUM, RFSUM, SISUM, HISUM, HFSUM
WRITE(6,5300) RIAVG, RFAVG, SIAVG, HIAVG, HFAVG
WRITE(6,5400) RISTD, RFSTD, SISTD, HISTD, HFSTD
5200 FORMAT(/15X,* TOTALS *, F9.4, F10.4, F14.4, 2F11.4)
5300 FORMAT(/15X,* AVGS. *, F9.4, F10.4, F14.4, 2F11.4)
5400 FORMAT(/15X,* STD. DEVN. *, F9.4, F10.4, F14.4, 2F11.4:///)
CALCULATE INITIAL AND FINAL ION FRACTIONS
XOD=SIAVG/1000.0
XND=XOD-(HFAVG-HIAVG)/1000.0
C=RIAVG
Y1D=RFAVG
CALCULATION OF EQUILIBRIUM CONSTANT = F(SOLN. NORMALITY)
EK=EQCONS(XOD)
RETURN
END
SUBROUTINE OUTPTCNC,NCYC,NPTS
COMMON /LAB1/ X(10,4),Y(10),D(4),A(4),DEL(4)
NCUS=NC-1
WRITE(6,650) NCUSP
6050 FORMAT(3*I,J25X,*CUSP NO. *CYCLE TRIAL X1
1 X2 X3 X4 Y
1/)

PRINT SUMMARY OF OPTIMIZATION EXPERIMENTS
WRITE(6,6100) NCYC,(X(1,I),I=1,4),Y(1),(A(K),K=1,4)
6100 FORMAT(15X,J5,* X*,5F10.4,4F8.4)
DO 10 J=2,NPTS
PP=J-1
WRITE(6,6300) J,PP
10 CONTINUE
RETURN
END

SUBROUTINE DELTACNPT, NPTS
COMMON /LAB1/ X(10,4),Y(10),D(4),A(4),DEL(4)
N=NPT-2
IF (Y(NPT)-Y(2)) 1,2,3
1 DEL(N)=-1.0
RETURN
2 DEL(N)=0.0
RETURN
3 DEL(N)=1.0
RETURN
END