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ENGINEERING AND ECONOMIC EVALUATION
OF COUNTERCURRENT AND FIXED BED ION
EXCHANGE PROCESSES

Technical Report Number 7

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Proface

The attached report is concerned with the relative costs, including buildings and all auxiliary equipment, of the countercurrent mixer-settler and moving bed processes and of the intermittent fixed bed processes for the separation and removal of ions, at concentrations up to 2500 ppm. It should be emphasized that such an engineering evaluation of the two countercurrent processes was not possible before design procedures were developed and tested as part of this project. The procedures presented in Technical Reports No. III, V, and VI, make it possible to predict the appropriate process to use for a given ion separation or removal problem, and to predict the optimum economic design of any one process.

The evaluation was based upon research data obtained on the separation of two alkali metal ions, but most of the qualitative conclusions would be valid for other ion systems. In brief these conclusions are:

1. There is a theoretical upper limit to the degree of separation which can be attained in a single countercurrent unit. For this reason, the fixed bed process, which has no such technical limitation, is more economical when the required degree of separation is greater than that attainable in a single countercurrent unit so that multiple moving bed units (cascades) are necessitated. However, when only one countercurrent unit is required to meet process specifications, the moving bed process is the most economical as the degree of separation approaches the theoretical limit. The moving bed process would be the most economical for ion separation over a wider range for more easily separated ions.
2. The moving bed process is universally more economical than the fixed bed for ion removal under both trace and gross conditions, and the savings are greater for gross than for trace removal.
3. The unit cost of ion removal by either the fixed bed or the moving bed process is reduced markedly when the throughput is increased, with the reduction greater for the already-favored moving bed.
4. The mixer-settler process is the most expensive for all commercial applications. This is due to the inherent complexity of multiple stages and valves and to the intermittent nature of the operations.

This report will be presented at the American Institute of Chemical Engineers national meeting at San Francisco, September 13-16, 1953, and will be submitted to Chemical Engineering Progress for publication.

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TABLE OF CONTENTS

	<u>Page</u>
ABSTRACT	1
INTRODUCTION	2
PROCESS SPECIFICATIONS	3
Performance Requirements	3
Design Data	4
Simplifying Assumptions	5
PROCESS DESIGN PROCEDURE	6
Mixer-Settler Process	6
Moving Bed Process	7
Fixed Bed Process	9
EFFECT OF PERFORMANCE REQUIREMENTS ON RELATIVE COSTS	10
Separation	10
Removal	11
General	12
CONCLUSIONS	12
ACKNOWLEDGMENTS	13
LITERATURE CITED	13
APPENDIX A - DESIGN OF A MIXER-SETTLER FOR SEPARATION OF TRACE IONS	A-1
Design of the Saturating Section	A-1
Design of the Eluting Section	A-3
Determination of Costs	A-3

TABLE OF CONTENTS (Continued)

	<u>Page</u>
APPENDIX B - DESIGN OF A MOVING BED	B-1
Separation of Trace Ions	B-1
Removal of a Trace Ion	B-3
Removal of a Gross Ion	B-3
APPENDIX C - DESIGN OF A FIXED BED	C-1
Separation of Trace Ions	C-1
Removal of a Trace Ion	C-5
Removal of a Gross Ion	C-6
APPENDIX D - NOMENCLATURE FOR APPENDICES A, B, AND C	D-1

LIST OF TABLES

	<u>Following Page</u>
TABLE I - TOTAL ANNUAL COSTS FOR MIXER-SETTLER SEPARATION PLANT	7
TABLE II - TOTAL ANNUAL COSTS FOR MOVING BED SEPARATION PLANT	8
TABLE III - TOTAL ANNUAL COSTS FOR FIXED BED SEPARATION PLANT	10
TABLE IV - COSTS OF REMOVAL PROCESSES	11
TABLE A-I - CAPITAL EQUIPMENT COSTS FOR MIXER-SETTLER SEPARATION PLANT	A-3
TABLE B-I - CAPITAL EQUIPMENT COSTS FOR MOVING BED SEPARATION PLANT	B-2
TABLE C-I - CAPITAL EQUIPMENT COSTS FOR FIXED BED SEPARATION PLANT	C-5

LIST OF FIGURES

	Following Page
Figure 1 - Flowsheet for a Mixer-Settler Process	6
Figure 2 - Number of Stages in Eluting Section of Mixer-Settler Unit as a Function of Elutant Flowrate	7
Figure 3 - Annual Cost Factors for Mixer-Settler Process as a Function of Elutant Flowrate	7
Figure 4 - Flowsheet for a Moving Bed Process	7
Figure 5 - Number of Transfer Units in Eluting Section of Moving Bed Unit and Total Annual Costs as a Function of Elutant Flowrate	8
Figure 6 - Flowsheet for a Fixed Bed Process	9
Figure 7 - Number of Transfer Units and Total Annual Costs of Fixed Bed Plant as a Function of Elutant Flowrate	9
Figure 8 - Comparison of Costs of Separation Plants	10
Figure 9 - Comparison of Elutant Acid Costs for Separation Plants	11
Figure B-1 - Design of Saturating Section of Moving Bed for Trace Ion Removal	B-3
Figure B-2 - Total Annual Costs of Moving Bed Trace Ion Removal Plants	B-3
Figure B-3 - McCabe-Thiele Diagram for Moving Bed Gross Ion Removal	B-3
Figure B-4 - Graphical Determination of Number of Transfer Units for Gross Ion Removal	B-4
Figure C-1 - Concentration Histories for Trace Ion Separation and Removal	C-1
Figure C-2 - Relations Among Fixed Bed Operational Parameters	C-2
Figure C-3 - Concentration History for Gross Ion Removal	C-6

1 June 26, 1953

Technical Report No. VII

ENGINEERING AND ECONOMIC EVALUATION OF MOVING
AND FIXED BED ION EXCHANGE PROCESSES

ABSTRACT

The paper describes a study undertaken to determine the most economical ion exchange techniques for the separation or removal of ionic components. Three modes of contacting the resin and solution are considered: in a fixed bed of resin; in an intermittent, mixer-settler type device; and in a continuous, countercurrent column.

The fixed bed operation involves the conventional procedure of cyclic saturation and elution of the stationary resin. The mixer-settler is composed of a number of equilibration stages and solution reservoirs and is divided into saturation and elution sections. The continuous, countercurrent column involves moving beds of resin in two columns, one for saturation and another for elution.

The only simple, yet precise, methods presently available for the design of all three processes apply to the exchange of trace components. The fixed bed techniques and calculation methods for trace exchange are reported in the literature. The design methods applying to the continuous column and mixer-settler have been previously described by the authors. In the case of the removal of gross ions, approximate procedures were used for the calculation of moving bed and fixed bed units.

The procedures are given by which the optimum economic design of each process was determined over a range of process specifications. Although these specifications are hypothetical, they were selected to provide realistic design problems. The relative costs of the processes including buildings and all auxiliary equipment, are presented for trace ion separation, trace ion removal, and gross ion removal; and the effect, on the cost, of product specifications and throughput is indicated.

INTRODUCTION

No reference has been found to any previous extensive comparative engineering analysis of fixed bed and countercurrent ion exchange processes, although Bauman (2) and Monet (9), among others, have discussed fixed bed costs. In fact, the state of engineering development, both theoretical and practical, has only recently reached a point which would permit the explicit design of each of the three processes considered.

Each of the processes, fixed bed, moving bed, and mixer-settler, utilized the properties of an ion exchange resin to bring about changes in the ionic composition of a solution. The exchange of one ion, say A, in solution with another, say G, in the resin can be represented by the equation

If another ion, say B, were also in solution, a similar mass action equation could be used to represent its exchange with ion G on the resin as

The separation of ions A and B by ion exchange is possible primarily because the equilibrium distribution between the resin and solution phases is different for each ion. In the fixed bed and moving bed processes, the differential diffusion rates of the ions through the resin particle, and through the solution film surrounding the particle, are also involved in the degree of separation.

Fundamentally then, all the processes are similar. In the separation of ions A and B in a feed stream, the solution would be contacted with G resin. The equilibria would favor the retention of one ion, say B, on the resin. Following saturation of the resin with feed solution, an elutant containing a high concentration of ion G would be contacted with the resin to displace ions A and B. In a removal process, the same mechanisms are involved, but they apply to the substantially complete removal of the specified ions in the feed rather than to their preferential fractionation.

The removal processes were evaluated in two areas of ion exchange--trace and gross conditions. The differentiation between the two conditions is not based on the absolute concentration of the ion concerned, but on its value relative to the total ionic level of the solution. A trace ion is dilute with respect to the total ionic concentration, whereas the concentration of a gross ion is of the same order of magnitude as, or (if it is the only ion present) is identical with, the total ionic concentration. Thus, gross ion A in one system can occur at the same absolute concentration as trace ion A in another system, if the total ionic level is much higher in the second case than the first.

The purpose of the economic evaluation was primarily to obtain relative cost information about the three ion exchange processes, so that an intelligent choice could be made in several areas of potential application.

PROCESS SPECIFICATIONS

Performance Requirements. Insofar as possible, the three ion exchange processes were compared on the basis of identical performance. Thus, the feed and effluent were completely defined with respect to composition and flowrate, and each process was designed to meet these requirements rigidly. Consequently, blending tanks were provided for the fixed bed plants to permit uniform composition of the effluent from this intermittent process, and holding tanks were provided for the mixer-settler plants to permit uniform flow of effluent from this intermittent process.

For the ion separation phase of the study, the feed contained two hypothetical trace ions, A and B, and a carrier elutant ion, G. Actually, the physical-chemical characteristics of this ion system were selected to correspond with the $\text{Li}^+ - \text{K}^+ - \text{H}^+$ ion system used for the experimental program described earlier (4-6). In effect then, ion A corresponded to Li^+ , B to K^+ , and G to H^+ . Since it was desired to make the evaluation as general as possible, the ions were designated A, B, and G, rather than as specific chemical entities.

The principal problem involved the separation of the trace ions A and B, and ion A was characterized as the more valuable component. For purposes of the study, the product effluent normally was required to contain ion A at 95% purity with respect to ion B. The specified recovery of ion A in the effluent was varied from 50% to 90% of that introduced in the feed. The performance requirements for the ion separation phase of the study are summarized below:

Feed: 5 gpm, $\text{A}^{\dagger} - 0.025 \text{ meq/ml}$, $\text{B}^{\dagger} - 0.025 \text{ meq/ml}$, $\text{G}^{\dagger} - 1.00 \text{ meq/ml}$.

Elutant: $\text{G}^{\dagger} - 1.05 \text{ meq/ml}$.

Effluent: 5 gpm at following purity-recovery combinations:

<u>Product</u>		<u>Processes Evaluated</u>		
<u>Purity</u>	<u>Recovery</u>	<u>Moving</u>	<u>Mixer-</u>	<u>Fixed</u>
<u>of A (%)</u>	<u>of A (%)</u>	<u>Bed</u>	<u>Settler</u>	<u>Bed</u>
95	50	X	X	X
"	60	X		X
"	70	X	X	X
"	74	X		
"	90	X		X
90	70	X		X

The evaluation was extended to two cases for the removal of ion B from a feed. The first concerned a feed containing trace ion B with G; in the other, the feed contained B alone. The effluent composition was specified to correspond with the amount of B in the product effluent from the ion separation plant at the 95% purity - 70% recovery level of A. For ion removal, only the moving bed and fixed bed processes were considered. The performance requirements are tabulated below:

Conditions	Feed			Product Effluent	
	B ⁺ meq/ml	G ⁺ meq/ml	Rate gpm.	Recovery Percent of B ⁺ in feed	
Trace	0.05	1.00	5	3.68	
	0.05	1.00	175	3.68	
Gross	0.05	0	175	3.68	

Design data. The data for the exchange of Li, K and H ions on Chempro C-20 cation resin have been presented in prior documents (4-6). These data were used for the current evaluation of ion exchange with hypothetical ions A, B, and G. The pertinent information is tabulated below:

Ultimate capacity of resin, $Q = 5.11$ meq/g oven dry.
 Density of resin, $\rho_p = 0.64$ g/cc true resin volume
 Void fraction of resin, $f_E = 0.39$
 Average wet particle diameter, $d_p = 0.07$ cm
 Trace distribution coefficients

$$M_{AG} = 0.87$$

$$M_{BG} = 3.30$$

Gross equilibrium constants

$$K_{AG} = 1.00$$

$$K_{BG} = 3.16$$

Counter-diffusivity coefficients

$$A^+ \text{ in solution, } (D_{LiH})_F = 1.85 \times 10^{-5} \text{ sq cm/sec}$$

$$B^+ \text{ in solution, } (D_{KH})_F = 2.18 \times 10^{-5} \text{ "}$$

$$A^+ \text{ in resin, } (D_{LiH})_P = 3.46 \times 10^{-6} \text{ "}$$

$$B^+ \text{ in resin, } (D_{KH})_P = 4.08 \times 10^{-6} \text{ "}$$

Density of solution, $\rho = 0.998$ g/cc

Viscosity of solution, $\mu = 0.0096$ poises

The height of a transfer unit for both the fixed bed and moving bed processes was determined from correlation relations (4,11) which involve the above data and the operating variables of the process.

The cations were assumed to be present as nitrates, so that stainless steel, glass-lined or rubber-lined equipment could be used for processing. The feed was assumed to contain radioactivity at such a level that the process would have to be operated by remote, automatic control from outside a concrete enclosure.

Wherever the costs of equipment or materials could be obtained from the current literature or suppliers' quotations, these figures were used. Otherwise, the costs of vessels and buildings were calculated by using estimated cost-per-square-foot factors.

It was necessary to recover the acid used for regenerating the resin in all the processes. Since it was outside the scope of the study to design a nitric acid recovery system, it was assumed that the acid cost (including recovery and make-up) would be one-quarter the market price at the time the study was made - \$0.0045/gal. of 1.05 N. This compares with a reported cost, for fuel alone, of \$0.0026/gal. for a single effect evaporator (1). Considering the nature of the material, the assumed recovery cost appears reasonable.

Simplifying assumptions. A number of assumptions were made to simplify the design of the various units. These are listed below:

1. One operator would be required per shift, for each process. Since all the processes would be provided with control devices, the labor requirements would probably be less (depending on the degree of automation), especially if the ion exchange unit were only part of a much larger operation.
2. After the cost of a typical plant of each type was determined, the standard scale-up factor of the 0.6 power of the size ratio was assumed for all costs except for labor and elutant, which were calculated directly.
3. The plants and equipment were amortized at 20% because of the corrosive nature of the solutions.
4. Within design accuracy, the contact efficiency in the mixer-settler was assumed to be 100% (complete equilibrium between phases at each contact).
5. The fixed bed units were assumed to contain two beds of resin to permit simultaneous saturation and elution.
6. Downflow of both saturant and elutant in the fixed bed was assumed because of the limitations of design theories presently available.

7. Upflow of both saturant and elutant in the moving bed was assumed because of mechanical and hydraulic problems involved in promoting upflow of resin and also because the design could be more firmly established on the basis of experimental experience.
8. Somewhat arbitrary, but thoroughly considered, cost factors were assigned to the maintenance of each type of process. The factors were based on the mechanical complexity, the ease of replacing parts, the surface area exposed to solution, and the amount of instrumentation involved.
9. In all the systems, the resin was eluted to convert 99.5% of its ultimate capacity to the G form prior to saturation. In the fixed bed, this exact concentration was encountered only at the effluent end of the column, all the rest of the resin being more completely eluted. This assumption was necessary in order to make use of simplified design equations, and it applied equally to all the processes.
10. The plants were assumed to operate on three shifts, 300 days per year.
11. All operations were carried out at 70°F.
12. The standard resin loss of 10% per year was assumed for the fixed bed, and 50% per year for the two countercurrent processes.

PROCESS DESIGN PROCEDURE

Mixer-Settler Process - A flowsheet of the proposed mixer-settler process is illustrated in Figure 1. It would involve a number of mixing tanks and solution reservoir tanks. Quantities of resin would be retained in the mixing tanks, and the solutions would be alternately equilibrated with the resin and transferred to the reservoirs. The plant would be operated so that, at any one time, part of the resin aliquots would be undergoing saturation while the others were being eluted. The method of operation would be exactly like that described in an earlier document (5), and the net result would be countercurrent contact between the resin and the solutions.

Process control would be vested in a cycle timer and a flame photometer. Holding tanks would be provided to smooth out the variable flow from the unit.

The design of the mixer-settler process for the separation of trace ions (the only cases considered for this unit) involved the design relations presented earlier. Sample calculations are given in Appendix A. The number of mixing stages and the quantity of resin in each stage was established for the saturating section, from the product specifications, and for each set of specifications there was only one particular combination of stages and resin aliquot volume per stage which would fulfill the requirements. Usually, of course, these calculations indicated something other than an integer number of stages, so the next larger integer was actually used. This meant the plant would produce a better separation than required, but this is one of the design limitations of this process.

The resin aliquots were retained in the mixing stages throughout all operations, so following each series of contacts with feed solutions a resin aliquot was subjected to a series of contacts with elutant solutions. Thus, the volume of resin in each stage was common to both the eluting and saturating sections. The conversion of the resin in the eluting section to 99.5% G form could be accomplished with an infinite number of designs -- from a large number of stages, each containing small elutant aliquots, to the converse -- each design based on elution of the more strongly held A ion. Any one of the combinations shown in Figure 2 would have resulted in the elution necessary for the 70% recovery, 95% purity plant, but the object was to find the optimum elutant flowrate.

A plant using one specific elutant flowrate (8 gpm) was completely designed, and the total annual cost for this plant was determined item-by-item. This cost estimation would be a report in itself, but the results are summarized in Table I.

Then, at each of the other combinations indicated in Figure 2, the total annual costs of the plant were calculated, using the standard scale-up factor of the 0.6 power of the size ratio for all costs except for labor and elutant. For this purpose, the size was based on the product of the number of stages and the volume of a mixer. The elutant consumption affected primarily the operating costs, and the number of stages the fixed charges. In Figure 3 the annual cost factors (derived from the data in Figure 2) are expressed as functions of the effective elutant flowrate. The design showing the lowest total annual cost was selected as the optimum. It will be noted that the optimum elutant flowrate was the 8 gpm at which the detailed cost calculations were carried out. This was more than coincidence, because preliminary cost studies enabled an estimation of the approximate optimum flowrate before the detailed costs were evaluated. In fact, such preliminary cost studies were made for the moving bed and fixed bed plants, as well, to aid in the selection of designs on which to make the more accurate, detailed cost studies.

In a like manner, a mixer-settler unit was designed to yield a product containing ion A at 50% recovery and 95% purity for which the costs were estimated by the 0.6 scale-up factor.

Moving Bed Process. The moving bed process was described in an earlier paper (4). Continuous, countercurrent contact between the resin and solution is accomplished in a cylindrical column containing a dense moving bed of resin. The resin flows by gravity down the column against a rising stream of solution. The resin is introduced into the top of the column by a resin transfer and dewatering valve, and is removed at the bottom by a corresponding valve.

The simplified flowsheet of the envisioned moving bed process is given in Figure 4. It would utilize two columns, one for saturation and the other for elution of the continuously flowing resin. The actual separation or removal of ions would take place in the saturation section, and the resin would be regenerated for recycling in the elution section.

Provision was made for automatic control through a continuous spectrophotometer with flame attachment on an effluent sidestream. This flame photometer would control the rate of resin transfer through the system. Fines would be removed continuously from the recycled resin to prevent carryover into the effluent. As was pointed out earlier, no specific provision was made for recovering the regenerant (ion G), but an adequate cost for such recovery was allowed.

In some respects the design approach approximated that used for the mixer-settler. The design method is outlined in detail in Appendix B. The design of the saturating section was fixed rather rigidly by the process specifications. First, the linear flowrate was limited below that which would carry resin out the top of the column, thus establishing the minimum column diameter for a given volumetric feed rate. The performance of the saturating column was specified in terms of the effluent composition, and there was only one resin flowrate which would result in this particular composition. This was found from the fundamental relations between the number of transfer units and the operating variables for the exchange of each ion of interest. These relations were presented in the earlier paper (4).

The design of the eluting column was somewhat more flexible, because the only requirement was that the resin be restored to 99.5% of its capacity in the G form. Since it was more difficult to elute ion B than A, it was necessary to design only for the exchange of ion B with the eluting ion, G. The one common factor between the two columns was the flowrate of the continuously recycled resin. Under these conditions, the desired elution could be obtained with an infinite number of specific combinations of elutant flowrates and number of transfer units, i. e., with a large number of transfer units (tall column) and a low elutant flowrate, or with a shorter column and higher volumetric rate. For instance, Figure 5 shows the relation between the number of transfer units and the elutant flowrate in the design of the eluting section of a unit to give 70% recovery and 95% purity of A in the product stream.

A plant utilizing the 8 gpm elutant flowrate was completely designed, and its cost was accurately determined. Table II gives a breakdown of the cost elements. The costs of the other moving bed plants were based on the 0.6 power of the size ratio (relative resin inventories) as compared with this design. The resultant annual costs, including labor and acid costs, are shown in Figure 5.

Similar sets of calculations were carried out for each of the product specifications listed in the Performance Requirements section. However, the design of the moving bed process for 90% recovery was more complex, because two cascades were required. It was thus necessary to establish first the optimum division of the load carried by each cascade and then the optimum design of the individual cascades.

In the design of a moving bed system for removal of trace ions, one has some freedom of choice in selecting the number of transfer units and resin flow rate for the saturating column. In practice, several reasonable combinations were selected, and for each such saturating column design, the optimum design of the eluting column was determined. Then the overall system showing the lowest annual cost was chosen. The design approach is also detailed in Appendix B.

The procedure used for evaluating the moving bed process for gross ion removal is a modification of that described in Reference (6). The required number of transfer units was found directly by a Chilton-Colburn graphical integration (3), based on a McCabe-Thiele type diagram for the binary system. A relation between gross and trace transfer heights developed by Vermeulen et al (11) and the moving bed correlation for trace transfer heights (4) were used to calculate the bed height from the number of transfer units. The design method is given in more detail in Appendix B, but the optimum system is chosen in the same manner as for trace ion removal. The costs for the optimum removal designs will be given in Table IV.

Fixed Bed Process. Figure 6 is a flowsheet of the proposed fixed bed process. The two resin beds would be alternately saturated with feed solution and eluted with G ion solution. Because of the transient concentration pattern of the effluent from the fixed beds, blending tanks would be provided to assure a uniform product from the unit. Diversion of the effluent from the ion exchange columns to the proper tanks, and of the feed and elutant to the proper beds, would be controlled automatically with a mechanism incorporating an integrating flame photometer. The control device would be complex, but it is in the realm of current instrumentation development.

Downflow of solution would be practiced, except for the occasional backwashing and simultaneous removal of fines, for which provision was made.

In most respects, the operation would resemble conventional fixed bed practice. Ion separation would occur largely as a result of the differential transfer of the individual ions down the column during elution.

The design of the fixed bed process for trace exchange followed the principles described by Vermeulen and Hiestor (10). By the procedure described in Appendix C, it was possible to calculate a number of specific fixed bed units which would meet each performance specification. Like the other two processes, if more resin (larger fixed beds) was used, less elutant was required. For the separation process involving a product with 70% recovery and 95% purity of A, the relation between the number of transfer units and effective elutant flowrate is shown in Figure 7.

As before, the plant based on the apparent optimum flowrate was selected (on the basis of prior cost studies) for a complete cost evaluation. Table III is a summary of the results of these detailed cost calculations for the 11.5 gpm fixed bed separation plant giving a recovery of 70% A in the product at a purity of 95%. The costs of the other designs shown in Figure 7 were derived by using the 0.6 scale-up factor based on the relative volumes of resin.

The removal of a trace ion by the fixed bed process can be accomplished with a multitude of specific combinations of number of transfer units (resin quantities), elutant quantities, and the ratio of product time to feed time. Since it is hard to conceive of a case where the product is not the treated feed, this ratio was taken as unity, and the resultant resin-elutant combinations were optimized on an economic basis. This procedure, nearly the same as that used for the separation design, is also described in Appendix C.

Although an exact method is available (7) for the design of a fixed bed process for gross ion removal, the amount of calculation required to optimize the design would have been excessive. For this reason the approximate, although reasonably accurate, procedure, based on the work described in reference (8) and outlined in Appendix C, was used to obtain the combinations of the number of transfer units and elutant ratios that would lead to the desired degree of ion removal. As before, the ratio of product time to feed time was taken as unity. The transfer units were converted to resin volumes by the method of Vermeulen et al (11), and the optimum cost combination was obtained in the usual manner. The costs for the optimum removal designs will be given in Table IV.

EFFECT OF PERFORMANCE REQUIREMENTS ON RELATIVE COSTS

Separation. In Figure 8 most of the results of the separation plant designs are presented graphically. The total annual costs are shown for each plant of optimum design as a function of recovery at a constant 95% purity of A in the product. Again, the reader should keep in mind that these costs should be viewed relative to each other and not as typical absolute costs for industrial plants.

Several important points stand out. The fixed bed process is the most economical except in the 59% to 74% recovery range, which favors the moving bed process. The limiting recovery obtainable with a simple countercurrent process, either moving bed or mixer-settler, is 74.7% for this particular ion-resin system. This limit is a function of the relative distribution coefficients of the ions (5).

As this maximum recovery is approached, the costs of such a process approach infinity. It is possible to obtain higher recoveries in a countercurrent process by passing the waste effluent through a second unit, or cascade. For instance, the moving bed process was designed with two cascades to obtain 90% recovery. The costs of such a complex system are intrinsically high, appreciably more than for a fixed bed process in the high recovery range.

The ion system used for this evaluation has a separation factor intermediate between the isotopes of one ion species, or like ions, on the one hand, and unlike ions or complexed systems, on the other. For the latter systems, which have high separation factors, the theoretical limit of recoveries attainable with a single countercurrent unit is much higher. Thus, the range of recoveries in which the moving bed system would be the most economical would be much larger for these systems.

It is interesting to note the effect of performance requirements on the costs of elutant acid (derived from the elutant consumption) as indicated in Figure 9. The data correspond with those represented in Figure 8. The direct effect of regenerant acid requirements on the total annual costs of a process are immediately evident. From this standpoint alone, the fixed bed shows the greatest economy except in the 63% to 74% recovery range.

The opposite slopes of the fixed bed and countercurrent curves are of interest. It is readily apparent that the elutant requirements should rise for the removal (and recovery) of more material from a fixed bed. However, in the case of the moving bed process, the recovery is a function of how much material in the feed passes through the saturating section without being attracted to the resin. Since less material would be picked up by the resin at high recoveries, less acid would be required to strip the resin in the eluting section.

The effects of changing the purity requirement from 95% to 90% at a constant recovery of 70% were evaluated for the fixed bed and moving bed process, and the comparative results are tabulated below.

<u>Cost Item</u>	<u>Fixed Bed</u>		<u>Moving Bed</u>	
	<u>Purity of A</u>		<u>Purity of A</u>	
	<u>90%</u>	<u>95%</u>	<u>90%</u>	<u>95%</u>
Annual Acid Cost	\$19,000	\$21,800	\$15,700	\$15,700
Total Annual Cost	63,500	70,000	55,000	57,150
Cost per 1000 gal.	29.40	32.40	25.40	26.50

It may be seen that the fixed bed costs are much more sensitive to purity specifications than the moving bed costs.

Removal. All the final cost results of the design calculations for ion removal systems are contained in Table IV. The costs of the ion separation processes corresponding to the 5 gpm trace removal processes are also tabulated for comparison purposes.

The results indicated that the ion removal cases considered favored the moving bed over the fixed bed process.* Moreover, it can be deduced

* This economy is obtained in spite of a higher transfer height for the moving bed unit than for the fixed bed, a factor which may be demonstrated by comparing the heights indicated by the correlations for the two processes (4,11) under similar operating conditions.

that, for removal, the moving bed would be more economical than the fixed bed throughout the entire range up to essentially 100% removal, because no cascading of the countercurrent units would be necessary. The costs of the equivalent trace separation and trace removal processes are identical for the moving bed and nearly so for the fixed bed.

As shown in Table IV, increasing the throughput requirement from 5 gpm to 175 gpm reduced the unit cost of trace ion removal by a factor of 3 for the fixed bed and by a factor of 4 for the moving bed. This means that the already favorable cost picture for the moving bed was improved by scale-up.

For either process, the annual cost for removal of a gross ion is very much less than for removal of a trace component. Moreover, the moving bed process is even more economical than the fixed bed under gross conditions than under trace.

General. It will be observed in Tables I, II and III that the annual resin replacement costs were a very small part of the total annual costs. Even higher resin losses than 50% could have been assumed for the countercurrent processes without affecting the results appreciably.

The two most important factors in the operating costs, labor requirements and acid recovery cost, may be lower in commercial application than is assumed for purposes of this evaluation.

It is anticipated that the labor cost for the moving bed system would be lower than for the fixed bed, because this is intrinsic in continuous processing. If, in practice, the labor requirements were less, it would enhance the already favorable economics of the moving bed method for ion removal. On the other hand, the over-all picture for ion separation would remain nearly unchanged; even if the moving bed process could be operated without any labor, the only effect would be a slight increase in the range of economical recoveries for the moving bed.

It is probable that the acid recovery costs would be lower in processing materials of commercial interest. If they were, the fixed bed would be favored, since the elutant cost is a greater proportion of the total cost for it than for the moving bed.

Thus, for commercial applications the reduced operating labor expected for the moving bed would be counterbalanced by the lower acid recovery costs favoring the fixed bed, and the relative economics of the two systems would still be substantially as indicated.

CONCLUSIONS

Although this quantitative information applies only to the specific ion-resin system considered, it would be possible to extrapolate the results, quantitatively, to related systems, especially for ion removal applications.

The principal qualitative conclusions of the evaluation are summarized below:

1. Except in a certain range of recoveries, the fixed bed process is the most economical for ion separation.
2. For ion removal, the moving bed process is more economical than the fixed bed process over the entire range of recoveries.
3. The unit cost of ion removal by either the moving bed or fixed bed process is reduced markedly when the flowrate is increased, with the reduction greater for the moving bed.

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14

TABLE I

TOTAL ANNUAL COSTS FOR MIXER-SETTLER SEPARATION PLANT-
70% RECOVERY, 95% PURITY OF A IN PRODUCT

OPERATING COSTS

Labor	\$15,100	
Supervisory and Clerical @ 10% Labor	1,510	
Payroll Overhead @ 20% Labor and Super.	3,320	
Utilities	690	
Resin	110	
Maintenance	8,270	
Acid	<u>15,700</u>	
Total Operating Costs		\$44,700

FIXED CHARGES

Taxes @ 2% Fixed Capital Investment*	3,330	
Insurance @ 1% Fixed Capital Investment	1,660	
Interest @ 6% " " "	9,980	
Depreciation @ 20% " " "	<u>33,300</u>	
Total Fixed Charges		<u>48,300</u>

TOTAL ANNUAL COSTS

\$93,000

* Fixed Capital Investment

Equipment	\$27,000	
Piping and Instrumentation	45,000	
Installation @ 35% first two items	25,200	
Building @ \$3.00/cu. ft.	12,000	
Auxiliary Facilities	<u>1,500</u>	
Total Physical Cost		\$110,700
Engr. and Constr. @ 35% Physical Cost		38,800
Contingencies @ 15% Physical Cost		16,600
Resin @ \$25.00/cu. ft.		<u>220</u>
Fixed Capital Investment		\$116,300

15

TABLE II

TOTAL ANNUAL COSTS FOR MOVING BED SEPARATION PLANT -
70% RECOVERY, 95% PURITY OF A IN PRODUCT

OPERATING COSTS

Labor	\$15,100	
Supervisory and Clerical @ 10% Labor	1,510	
Payroll Overhead @ 20% Labor and Super.	3,320	
Utilities	850	
Resin	180	
Maintenance	6,590	
Acid	<u>15,700</u>	
Total Operating Costs		\$43,250

FIXED CHARGES

Taxes @ 2% Fixed Capital Investment*	960	
Insurance @ 1% Fixed Capital Investment	480	
Interest @ 6% " " "	2,870	
Depreciation @ 20% " " "	<u>9,560</u>	
		<u>\$13,900</u>

<u>TOTAL ANNUAL COSTS</u>	<u>\$57,150</u>
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* Fixed Capital Investment

Equipment	\$10,400	
Piping and Instrumentation	9,820	
Installation @ 35% first two items	7,100	
Building @ \$3.00/cu. ft.	2,700	
Auxiliary Facilities	<u>1,500</u>	
Total Physical Cost		\$31,600
Engr. and Constr. @ 35% Physical Cost		11,100
Contingencies @ 15% Physical Cost		4,700
Resin @ \$25.00/ cu. ft.		<u>360</u>
Fixed Capital Investment		<u>\$47,800</u>

TABLE III

TOTAL ANNUAL COSTS FOR FIXED BED SEPARATION PLANT -
70% RECOVERY, 95% PURITY OF A IN PRODUCT

OPERATING COSTS

Labor	\$15,100	
Supervisory and Clerical @ 10% Labor	1,510	
Payroll Overhead @ 20% Labor and Super.	3,320	
Utilities	820	
Resin	30	
Maintenance	4,670	
Acid	22,200	
Total Operating Costs		\$47,700

FIXED CHARGES

Taxes @ 2% Fixed Capital Investment*	1,540	
Insurance @ 1% Fixed Capital Investment	770	
Interest @ 6% " " "	4,720	
Depreciation @ 20% " " "	15,400	
Total Fixed Charges		\$22,400

TOTAL ANNUAL COSTS \$70,100

* Fixed Capital Investment

Equipment	\$17,550	
Piping and Instrumentation	13,200	
Installation @ 35% first two items	10,800	
Building @ \$3.00/cu. ft.	8,100	
Auxiliary Facilities	1,500	
Total Physical Cost		\$51,150
Engr. and Constr. @ 35% Physical Cost		17,900
Contingencies @ 15% Physical Cost		7,700
Resin @ \$25.00 cu. ft.		300
Fixed Capital Investment		\$77,050

TABLE IV

COSTS OF REMOVAL PROCESSES

A - FIXED BED PROCESSES

<u>Cost Item</u>	<u>Trace Separation</u> 70% Recovery of A 95% Purity of A @ 5 gpm	<u>Trace Removal</u> 3.68% Recovery of B @ 5 gpm	<u>Trace Removal</u> 3.68% Recovery of B @175 gpm	<u>Gross Removal</u> 3.68% Recovery of B @175 gpm
Annual Acid Costs	\$21,800	\$22,600	\$558,000	\$150,000
Total Annual Costs	70,000	68,500	760,000	214,000
Cost per 1000 gal.	32.40	31.70	10.00	2.83

B - MOVING BED PROCESSES

<u>Cost Item</u>	<u>Trace Separation</u> 70% Recovery of A 95% Purity of A @ 5 gpm	<u>Trace Removal</u> 3.68% Recovery of B @ 5 gpm	<u>Trace Removal</u> 3.68% Recovery of B @175 gpm	<u>Gross Removal</u> 3.68% Recovery of B @175 gpm
Annual Acid Costs	\$15,700	\$15,500	\$376,000	\$ 47,000
Total Annual Costs	57,150	57,150	509,000	108,000
Cost per 1000 gal.	26.50	26.50	6.70	1.42

18

FIG. I
FLOWSHEET FOR A MIXER-SETTLER PROCESS

A-337-TR7-96

FIG. 2
NUMBER OF STAGES IN ELUTING SECTION OF MIXER-SETTLER UNIT
AS A FUNCTION OF ELUTANT FLOWRATE
70% R AND 95% p

A-337-TR7-102

FIG. 3
ANNUAL COST FACTORS FOR MIXER-SETTLER PROCESS
AS A FUNCTION OF ELUTANT FLOWRATE
70% R AND 95% p

A-337-TR7-103

The diagram illustrates a resin recycling system with the following components and flow paths:

- RESIN RESERVOIR:** A funnel-shaped tank at the top left that feeds into a square processing unit.
- RESIN VALVE:** A square valve located between the Resin Reservoir and the Saturating Column.
- SATURATING COLUMN:** A funnel-shaped column in the center that receives 'FEED' from the right and resin from the Resin Reservoir. It feeds into another square processing unit.
- ELUTANT TANK:** A rectangular tank on the left that receives 'WASTE' and feeds into a square processing unit.
- RECYCLE:** A line from the bottom of the Elutant Tank's processing unit, passing through a valve 'S', that feeds back into the Saturating Column.
- RESIN VALVE:** A square valve located between the Saturating Column and the Eluting Column.
- ELUTING COLUMN:** A funnel-shaped column at the bottom right that receives 'ELUTANT' from the right and resin from the Saturating Column. It feeds into a square processing unit.
- SURGE TANK:** A small rectangular tank between the Eluting Column and the Resin Surge Tank.
- RESIN VALVE:** A square valve located between the Surge Tank and the Resin Surge Tank.
- RESIN SURGE TANK:** A small rectangular tank at the bottom right that feeds back into the Eluting Column.
- RECYCLE:** A line from the bottom of the Eluting Column's processing unit, passing through a valve 'S', that feeds back into the Elutant Tank.
- WASTE:** A line from the left side of the Elutant Tank's processing unit that exits the system.
- RESIN RECYCLE:** A line from the bottom of the Eluting Column's processing unit, passing through a valve 'S', that feeds back into the Resin Reservoir.
- PRODUCT TANK:** A rectangular tank at the top right that receives 'PRODUCT' from the right and resin from the Resin Reservoir. It feeds into a square processing unit.
- PRODUCT:** A line from the bottom of the Product Tank's processing unit, passing through a valve 'S', that exits the system.

A-337-TR7-95

FIG. 5
 NUMBER OF TRANSFER UNITS IN ELUTING SECTION
 OF MOVING BED UNIT AND TOTAL ANNUAL COSTS
 AS A FUNCTION OF ELUTANT FLOWRATE
 70% R AND 95% p

A-537-TR7-101

FIG. 6
FLOWSHEET FOR A FIXED BED PROCESS

A-337-TR7-94

FIG. 7

NUMBER OF TRANSFER UNITS AND TOTAL ANNUAL COSTS OF
FIXED BED PLANT AS FUNCTION OF ELUTANT FLOW RATE
70% R AND 95% p

B-CU337-82

FIG. 8

COMPARISON OF COSTS OF
SEPARATION PLANTS
95% PURITY OF A IN PRODUCT

A-CU337-77

FIG. 9
COMPARISON OF ELUTANT ACID COSTS FOR
SEPARATION PLANTS
95% PURITY OF A IN PRODUCT

A-CU337-79

3. Substitution in Equation (5-23) leads to

$$\frac{\ln \left[\frac{E_A - 0.30}{E_A - (1 - 0.30)} \right]}{\ln E_A} = \frac{\ln \left[\frac{\delta E_A - 0.963}{\delta E_A (1 - 0.963)} \right]}{\ln \delta E_A}$$

and the trial-and-error solution for E_A indicates

$$E_A \approx 0.300 \text{ (theoretical limiting value)}$$

$$E_B \approx (3.79)(0.300) = 1.137$$

4. Then the number of contacts was established from E_B

$$N_C = \frac{\ln \left[\frac{1.137 - 0.963}{1.137 (1 - 0.963)} \right]}{\ln 1.137} = 11.24$$

and since two contacts took place per physical stage, six mixing stages were required for the saturating section.

5. The effective resin flowrate was determined

$$E_A = \frac{K_{AG} W_P Q}{V_F C_s} \quad (5-43)$$

$$W_P = \frac{E_A V_F C_s}{K_{AG} Q}$$

$$5 \text{ gpm} = 315 \text{ cc/sec}$$

$$\begin{aligned} W_P &= \frac{(0.300)(315)(1.05)}{(0.87)(5.11)} = 22.4 \text{ g resin/sec} \\ &= 0.120 \text{ cu.ft./min} \end{aligned}$$

6. Each operating cycle involved two resin-solution contacts of 1.5 minutes and six transfers of 0.5 minutes; thus the cycle time was

$$(2)(1.5) + (6)(0.5) = 6 \text{ min}$$

7. Since the operation was such that two contacts were equivalent to one physical mixing stage, the quantity of resin per stage was calculated from the effective flowrate and the cycle time,

$$(0.120)(6) = 0.723 \text{ cu.ft./stage}$$

APPENDIX A

DESIGN OF A MIXER SETTLER FOR SEPARATION OF TRACE IONS

Design of the Saturating Section

1. There is only one combination of the dimensionless operating factor E^* for each ion and the number of equilibrium contacts which will allow the attainment of any specified separation. For ion A, the number of contacts is defined by

$$(N_C)_A = \frac{\ln \left[\frac{E_A - \gamma_A}{E_A(1 - \gamma_A)} \right]}{\ln E_A} \quad (5-8A)**$$

and an exact correlary defines $(N_C)_B$

$$(N_C)_B = \frac{\ln \left[\frac{E_B - \gamma_B}{E_B(1 - \gamma_B)} \right]}{\ln E_B} \quad (5-8B)$$

However,

$$(N_C)_A = (N_C)_B \quad (5-22)$$

$$\frac{E_B}{E_A} = \frac{K_{BG}}{K_{AG}} \quad E_A = \delta E_A = 3.79 E_A$$

and thus

$$N_C = \frac{\ln \left[\frac{E_A - \gamma_A}{E_A(1 - \gamma_A)} \right]}{\ln E_A} = \frac{\ln \left[\frac{\delta E_A - \gamma_B}{\delta E_A(1 - \gamma_B)} \right]}{\ln \delta E_A} \quad (5-23)$$

2. Here γ is the approach to equilibrium transfer achieved for the designated ion, and this approach is established for each ion from the recovery and purity specifications. For the product specifications of 70% recovery and 95% purity of A in the product, the approaches to equilibrium would be:

$$\gamma_A = 1 - R_A = 1 - 0.70 = 0.30 \quad (5-20)$$

$$\gamma_B = 1 - \frac{R_A}{P_A / (1 - P_A)} = 1 - \frac{0.70}{0.95/0.05} = 0.963 \quad (5-21)$$

* The definitions of all symbols are given in Appendix D.

** This number indicates first the reference according to the Literature Cited and, second, the equation number in that reference.

Design of the Eluting Section

1. The eluting section was designed for the retention of only 0.5% of the resin capacity for ion B. In terms of the dimensionless operating factors,

$$\frac{Y_B}{E_B} = 0.995$$

2. The concentration of the elutant was identical with the total concentration of the saturant, and the effective resin flowrate was identical for both sections, so the effective elutant flowrate was inversely proportional to the E_B value used in the eluting section.

$$\begin{aligned} \text{Effective Elutant Flowrate} &= 5 \text{ gpm} \frac{(E_B)_{\text{sat}}}{(E_B)_{\text{elut}}} \\ &= \frac{(5)(0.300)}{(E_B)_{\text{elut}}} \end{aligned}$$

3. A number of E_B values, each associated with a different solution flowrate, were assumed. For each, a specific number of contacts gave the desired degree of elution from Equation (5-8B). The inverse proportionality relation between the number of stages in the eluting section and the effective elutant flowrate was indicated in Figure 5.

Determination of Costs

1. A complete flowsheet of the mixer-settler process was prepared for a plant involving twelve mixing stages and an effective elutant flowrate of 8 gpm. It was similar to, but more detailed than, Figure 4 in the body of the paper.

2. The volumes of the mixers, settlers, and accessory tanks were determined from the volumes of the solution and resin aliquots. The economic pipe diameters and their lengths were determined. Blowers, agitators, pumps, and classifiers of suitable design were selected. Appropriate motorized valves and control devices were designated. The size of the building was based upon the most compact plant layout possible with allowances for replacement and maintenance of equipment. The detailed costs of the equipment, piping, and instrumentation are given in Table A-I.

3. The annual acid costs were calculated directly from the 8 gpm of elutant. These and the equipment costs were summarized in Table I.

4. The costs for the other elutant flowrates were determined for all factors except labor and acid by using the 0.6 power of the size ratios, based on the product of the number of stages and the volumes of the mixers. The labor cost was a constant, and the acid cost was calculated for each specific flowrate.

TABLE A-I

30

CAPITAL EQUIPMENT COSTS FOR MIXER-SETTLER
SEPARATION PLANT
70% RECOVERY, 95% PURITY OF A IN PRODUCT

<u>Item</u>	<u>Size</u>	<u>Material</u>	<u>No</u>	<u>Unit</u>	<u>Total</u>
			<u>Reqd</u>	<u>Cost</u>	<u>Cost</u>
Equipment					
Mixers	1'6"D x 5'3"	glass lined	12	\$670	\$8040
Reservoirs	1'6"D x 4'9"	304 stainless	12	400	4800
Feed Surge Tank	2'D x 2'6"	" "	1	350	350
Elutant Surge Tank	2'D x 4'3"	" "	1	550	550
Product Blending Tank	4'D x 4'	" "	1	1400	1400
Spent Elutant Tank	4'D x 6'	" "	1	1800	1800
Metering Pumps		" "	2	1000	2000
Diaphragm Pump		glass lined	1	1000	1000
Centrifugal Pump		304 stainless	1	350	350
Misc. Accessory Equipment					6700
					\$26,990
Piping and Instrumentation					
Pipe and Fittings		304 stainless			14,500
Motor Gate Valves	2 $\frac{1}{2}$ "	" "	52	250	13,000
Motor 3-way Valves	2 $\frac{1}{2}$ "	" "	24	350	8,400
Motor 3-way Valves	3 $\frac{1}{2}$ "	" "	2	550	1,100
Level Controls			12	175	2,100
Sight Glasses			12	75	900
Cycle Timer			1	500	500
Photometer Controller			1	4500	4,500
					\$45,000

DESIGN OF A MOVING BED

Separation of Trace IonsA. Design of the Saturating Section

1. Like the mixer-settler design described in Appendix A, only one specific combination of the number of transfer units, Nt ,* and the dimensionless operating factor, E , will give the desired approach to equilibrium transfer, γ , for each ion in a separation problem. For ion A, this relation is

$$(Nt)_A = \frac{E_A}{1-E_A} \cdot \ln \left[\frac{E_A (1-\gamma_A)}{E_A - \gamma_A} \right] \quad (4-14A)**$$

and a similar expression applies to ion B.

2. However, the number of transfer units is not identical for both ions being separated, the common factor between the two being the height of the moving resin bed. Thus, it is necessary to know the relative transfer heights for each trace ion. Under the operating conditions studied experimentally, the following correlation was found between the height of a transfer unit and the operating variables and the system constants:

(4-37')

$$\left[\frac{h}{(Nt_A)_{OFdp}} \right] = \frac{1.6}{f_E (Re \cdot Sc)^{0.5}} \left[\frac{d_p R_F}{6 (1-f_E) S(D_A)_F} \right] + \frac{1}{E_A} \cdot \frac{0.6}{(1-f_E)} \left[\frac{d_p R_P}{6 (1-f_E) S(D_A)_P} \right]$$

From an evaluation of this relation at the maximum linear solution velocity which experiments showed would not cause resin carryover (4), the relative transfer heights for ions A and B were established. This led to the ratio of the number of transfer units for the ions, which is the inverse of the ratio of the transfer heights.

3. Then, by an approach analogous to that used in Appendix A, the equations expressing the respective Nt values were related according to the ratio of $(Nt)_A : (Nt)_B$ as follows,

$$\frac{(Nt)_B}{(Nt)_A} \cdot \frac{E_A}{1-E_A} \cdot \ln \left[\frac{E_A (1-\gamma_A)}{E_A - \gamma_A} \right] = \frac{\delta E_A}{1-\delta E_A} \cdot \ln \left[\frac{\delta E_A (1-\gamma_B)}{\delta E_A - \gamma_B} \right]$$

and the E_A value was found by trial and error. The number of transfer units

*The definitions of all the symbols are given in Appendix D.

**This number indicates, first, the reference according to the Literature Cited and, second, the equation number in that reference.

for each ion was determined by Equation (4-14).

5. The column diameter was selected from the linear solution velocity (0.46 ft/min) and the volumetric flowrate of the solution. With E_A and the solution flowrate, R_F , established, the required resin flowrate, R_P , was calculated:

$$E_A = \frac{M_{AG} R_P}{R_F C_L} \rho_P Q \quad (4-16A)$$

This completed the design of the saturating column.

B. Design of the Eluting Section

1. The eluting section was designed for the retention of only 0.5% of the resin capacity for ion B, and the design approach was identical with that used for the mixer-settler.

2. The elutant flowrate was related to the feed flowrate by the ratio of $(E_B)_{sat}$ to the unknown $(E_B)_{elut}$.

3. Again a number of $(E_B)_{elut}$ values were assumed, each giving a specific number of transfer units and an elutant flowrate, as indicated in Figure 5 in the body of the paper.

C. Determination of Costs

1. A detailed plant layout, more complete than that shown in Figure 4, was prepared for one plant design utilizing a specific intermediate elutant flowrate, and the equipment was sized.

2. The individual equipment costs were determined and are given in Table B-I. Then the annual acid costs were established from the elutant flowrate, and all the annual cost factors were summarized in Table II.

3. The costs of the plants utilizing the other elutant flowrates were determined with the 0.6 power of the size ratios, based on the total volumes of the two resin beds. As before, the labor cost was constant and the elutant costs were calculated directly for each elutant flowrate.

D. Cascading

To achieve the 90% recovery of ion A with the moving bed process, it was necessary to cascade two units. For this design, three arbitrary divisions of the load between the two units were selected. Then the optimum design of each of the six units was determined by the procedure described

* The principles of cascading are discussed in reference (5).

above. The over-all design giving the lowest annual cost for the two-unit combination was chosen as the most economical.

Removal of a Trace Ion

1. Unlike the rigid design of the saturating section called for by an ion separation problem, the design for ion removal is flexible from a technical viewpoint. An infinite number of combinations of the number of transfer units and the resin flowrate will give the desired removal.

2. As before, the linear flowrate, and consequently the column diameter, was fixed by hydraulic limitations.

3. The four $(Nt)_{B-E_B}$ combinations indicated in Figure B-1 were found by Equation (4-14B) to give a recovery (leakage) of 3.68% of ion B in the product. The resin flowrate was directly proportional to E_B .

4. For each saturating section design, the eluting section could again have an infinite number of designs for restoring the resin to 99.5% acid form. Four specific elution designs were selected for each of the four saturating section designs.

5. The resultant total annual costs of all sixteen designs were obtained by applying the scale-up factor with the previously calculated separation plant cost. Figure B-2 shows these costs as functions of the respective E_B values, where $(E_B)_{elut}$ is inversely proportional to the elutant flowrate.

6. The minimum points of the curves were connected with the dashed line, the minimum of which indicated the optimum design.

Removal of a Gross Ion

1. The equilibrium curve or McCabe-Thiele y-x diagram, Figure B-3, was prepared for component B from the equilibrium relation

$$K_{BG} = \frac{y_B x_G}{x_B y_G} \quad (6-5B)$$

Since for a two component system $x_G = 1 - x_A$ and $y_G = 1 - y_A$, this leads to

$$x_B = \frac{y_B}{K_{BG} + y_B (1 - K_{BG})}$$

where $K_{BG} = 3.16$.

2. A suitable operating line for the saturating section was drawn in. The lower end of the line was fixed at $y_{entr} = 0.005$, $x_{exit} = 0.0368$; since it was required that the feed resin be 99.5% eluted, and the effluent solution composition was fixed by the 3.68% recovery. The upper end of the line was fixed with respect to the feed composition, $x_{entr} = 1.00$, but the corresponding effluent resin composition was a variable for each design; in this instance, the B content of the effluent resin was taken as $y_{exit} = 0.70$. The slope of this operating line ($R_{FC}/R_p/Q = 0.722$) is the ratio of the equivalent flowrates of the solution and resin phases. The solution flowrate is fixed by the feed rate, and thus the slope determines the resin flowrate.

3. Then the transfer unit diagram, Figure B-4, was constructed according to the displacement between the equilibrium and operating lines across the y-x diagram. The area under this curve and between x_{entr} and x_{exit} gives the number of transfer units, $(Nt)_{OF} = 4.27$, involved in the exchange, if diffusion through the fluid film is the rate-controlling mechanism. However, experimental work (6) has shown that under the proposed operating conditions, internal diffusion also exerts a rate-controlling effect. This problem was resolved by using the correlation of Vermeulen et al (11) to obtain the ratio of the internal to external effects at the specific flow rates involved.

This ratio was then used to correct the number of transfer units to the number represented by the combined film diffusion transfer rates.

4. Then the bed height for the saturating section was calculated, using the moving bed Ht correlation (4).

5. For this saturating section design, three eluting section designs were considered. These are represented by the three operating lines above the equilibrium line in Figure B-3. It will be noted that at the lower terminus of these lines the y_{exit} for the eluting section is equivalent to the y_{entr} for the saturating section, since the resin is recycled between sections. Also, $(y_{entr})_{elut} = (y_{exit})_{sat}$. The slopes of the lines are equal to the ratio of the equivalent elutant flowrate to the equivalent resin flowrate. The latter was fixed by the saturating section design, so that each curve represented a different elutant flowrate.

6. As in step 3, a Chilton-Colburn diagram was prepared for each eluting section operating line, and the corrected number of transfer units was determined as before.

7. As in the previous cases, the required number of transfer units was larger for low elutant flowrates.

8. The entire design procedure was repeated for two other saturant designs (operating lines), each with three eluting section designs.

9. The over-all design showing the lowest total annual costs (obtained by the procedures described in the trace separation discussion) was selected as the optimum.

TABLE B-1

35

CAPITAL EQUIPMENT COSTS FOR MOVING BED
SEPARATION PLANT

70% RECOVERY, 95% PURITY OF A IN PRODUCT

<u>Item</u>	<u>Size</u>	<u>Material</u>	<u>No.</u> <u>Reqd.</u>	<u>Unit</u> <u>Cost</u>	<u>Total</u> <u>Cost</u>
Equipment					
Saturation Column	1'6"Dx6'	glasslined	1	\$650	\$650
Elution Column	2' Dx5'	" "	1	760	760
Surge Tanks	1' Dx1'	304 stainless	5	70	350
Resin Reservoir	1' Dx2'	glasslined	1	160	160
Resin Surge Tank	1' Dx2'	" "	1	160	160
Resin Valves		304 stainless	3	1000	3000
Diaphragm Pump		glasslined	1	1000	1000
Centrifugal Pump		304 stainless	6	350	2100
Misc. Accessory Equipment					<u>2300</u>
					\$10,480

Piping and Instrumentation

Pipe and Fittings		304 stainless			1850
Motor Gate Valves	1"	" "	11	90	990
Flow Control Valves	1"	" "	2	200	400
Flow Controllers	1"	" "	2	600	1200
Level Controls			5	175	875
Photometer Controller			1	4500	<u>4500</u>
					9815

FIG. B-1

DESIGN OF SATURATING SECTION OF MOVING BED FOR
TRACE ION REMOVAL

337-TR7-97

FIG. B-2

TOTAL ANNUAL COSTS OF MOVING
BED TRACE ION REMOVAL PLANTS

A-337-TR7-100

38

FIG. B-3

McCABE-THIELE DIAGRAM FOR
MOVING BED, GROSS ION REMOVAL

A-337-TR7-98

FIG. B-4
GRAPHICAL DETERMINATION OF NUMBER OF
TRANSFER UNITS FOR GROSS ION REMOVAL

A-337-TR7-99

DESIGN OF A FIXED BED

The operation of a fixed ion exchange bed can be broken down into two basic steps, saturation and elution (or regeneration), the periods of which are not necessarily equal. The introduction of the feed and elutant solutions coincide with these two steps. The effluent solution is likewise divided into two portions, a product or treated solution and a waste solution.

In removal processes the treated solution is usually the feed that has passed through the column, and the waste is the spent elutant. However, for separations, this is not necessarily so, since then the purpose of the elutant is to assist in separating the ions in their passage down the column, and therefore the point at which the partially separated ions are divided into a product solution and a waste solution may not occur at the cessation of feed solution passage.

This is shown more clearly in Figure C-1, which is a depiction of the effluent concentrations from a fixed bed. The top diagram shows the separation of ions A and B, with the product solution containing most of the ion A fed, contaminated with only a small amount of ion B. Note that, as suggested earlier, the feed solution and product solution periods do not cease at the same time. The bottom diagram concerns the removal of ion B from the feed to give a treated solution containing only a small amount of it. Although the two diagrams represent the same fraction of B in the product solution from both processes, the two curves for ion B are not identical due to differences in the operational parameters.

Separation of Trace Ions

A. Design

1. Figure C-1 is drawn by the evaluation of the concentration history equations of Vermeulen and Hiester (10) at constant s .*

$$\text{for } t \leq T, \frac{x_A}{(x_A)_0} = J(s_A, t_A) \quad (10-19a)**$$

$$\text{for } t \geq T, \frac{x_A}{(x_A)_0} = J(s_A, t_A) - J(s_A, t_A - T_A) \quad (10-20)$$

* The definitions of all symbols are given in Appendix D;

** This number indicates, first, the reference according to the Literature Cited, and, second, the equation number in that reference.

where

$$J(u, v) = 1 - \int_0^u e^{-v - \xi} I_0(2\sqrt{\xi v}) d\xi \quad (10-19b)$$

In the figure the total amounts of ion A and of ion B fed to the column during a cycle are represented by the areas under their respective concentration curves. In the upper diagram the recovery of A in the product solution thus becomes

$$R_A = \frac{\text{Area I} + \text{Area II}}{\text{Area I} + \text{Area II} + \text{Area III}}$$

and the purity

$$P_A = \frac{\text{Area I}}{\text{Area I} + \text{Area II}}$$

For ion removal of B, forgetting ion A, the degree of removal is given by

$$1 - \frac{\text{Area II}}{\text{Total Area for Ion B}}$$

In mathematical terms R_A would then be

$$R_A = \frac{\int_0^{t_1} \left[\frac{X_A}{(X_A)_0} \right] dt}{\int_0^{\infty} \left[\frac{X_A}{(X_A)_0} \right] dt} = \frac{\int_0^{t_1} \left[\frac{X_A}{(X_A)_0} \right] dt}{T}$$

The integral cannot be solved explicitly in terms of s , and T , however, so it has to be evaluated graphically by planimetering the areas under curves in plots like Figure C-1. This was done for a large number of combinations of these operational parameters and the results were expressed as shown in Figure C-2. Similar plots were also prepared for the other product specifications.

2. As with the moving bed, the number of fixed bed transfer units, s_A , were not identical for both ions A and B, the common factor between the two being the height of the resin column. The ratio of s_A to

to s_B would thus be inversely proportional to the relative transfer heights for the two ions. This can be obtained from the correlation between the height of a fixed bed transfer unit and the operating variables and system constants.

$$\left[\frac{h}{s_A d_p} \right] = \frac{0.29}{f_E (Re \cdot Sc)^{0.5}} \left[\frac{d_p R}{6(1-f_E)S(D_A)_F} \right] + \frac{1}{D_A} \cdot \frac{0.04}{f_E} \left[\frac{d_p R}{6(1-f_E)S(D_A)_F} \right] \quad (4-34')$$

The solution linear flowrate used in these calculations was taken in all cases to be the same as that used in the moving bed calculations.

3. The point at which elution is completed (represented by the dimensionless parameter t_2) is dependent on the length of the resin bed (represented by the dimensionless parameter s). The amount of elutant per unit of feed (represented by $\frac{t_2-T}{T}$) is dependent on the amount of feed solution (T). It is therefore necessary to find the bed length and elutant combinations that will give the proper product specifications, and then to determine the optimum combination. This was done in the following manner:

- a. The ratio of top product to feed charge (t_1/T) is identical for both ions.
- b. The ratio of the feed charge to bed length fraction (T/s) for the two ions is the separation factor δ , i.e.

$$\frac{(T/s)_A}{(T/s)_B} =$$

- c. At a given t_1/T , there is only one value of T_A/s_A for all values of $s_A > 4$. Thus, there is only one value of T_B/s_B , $= (T_A/s_A)/\delta$ as indicated by the dotted line on Figure C-2, for all values of s_A . This dotted line corresponds to a specific value of s_B for the given t_1/T . Knowing both T_B/s_B and s_B for this t_1/T ratio, T_B can be calculated for the product specifications indicated.
- d. From this s_B value and the evaluation of the equation for resin concentrations at the completion of elution, i.e. at 99.5% eluted:

$$\frac{y_B}{(y_B)_0} = J(t_{R2} - T_B, s_B) - J(t_{R2}, s_B) \approx 0.005 - 0,$$

the value of t_{R2} can be calculated.

- e. This was repeated for series of t_1/T values to obtain a set of bed length - elutant volume combinations;

at $R_A = 70\%$ and $P_A = 95\%$

t_1/T	0.96	1.0	1.03	1.1	1.2
s_B	120	48	32	20.5	14.5
T_B	120	41	24.6	13.0	7.4
t_{B2}	284	118	81	54	39.5
$\frac{t_{B2}-T_B}{T_B}$	1.37	1.88	2.30	3.15	4.35

The last item is directly the gallons of elutant per gallon of feed as long as their flowrates and total concentrations are equal, as has been assumed. The plot of transfer units as a function of elutant flow rate shown in Figure 7 has been based on this information.

f. It is conventional practice to have normally two resin columns, one being saturated while the other is being eluted. However, in those cases where the elution time is greater

than the feed time i.e., $\frac{t_{B2}-T_B}{T_B} > 1$ there is an overlapping of the elution step which prevents uninterrupted treatment of the feed. Then, to have an effective feed flowrate of 5 gpm at all times, the actual solution flowrate must be increased as follows

$$R = \frac{1}{2} \left(\frac{t_{B2}}{T} \right) (5) \text{ gpm}$$

The increase in the actual solution flowrate is reflected in an increase in the cross sectional area of the column, S , since the linear flowrate must remain constant. Likewise the effective elutant flowrate is 5 gpm increased by the ratio of the elutant time to feed time.

$$E = \frac{t_{B2} - T_B}{T_B} (5) \text{ gpm}$$

This rate is important since it determines the cost of elutant per year.

4. The total volume of resin associated with each value of s_B can now be calculated from the bed length, indicated by the correlation (Equation (4-34)), the column area S , and the fact that two beds were used. The actual cycle times can be calculated from the T/s and the t/T ratios since

$$T/s = \frac{\tau_{\text{feed}}}{Dh Sfe/R}$$

$$t/T = \tau/\tau_{\text{feed}}$$

B. Determination of Costs

1. A complete flowsheet of the fixed bed process was prepared for a design involving an s_B of 32 and an effective elutant flowrate of 11.5 gpm. It was similar to, but more detailed than, Figure 6 in the body of the paper.

2. The volumes of the resin tanks were determined from the resin requirements, and the surge tanks were sized from the cycle times, effective solution flowrates, and actual flowrates through the fixed bed. The blending tanks were likewise designed to hold the complete product for one cycle from one resin column. The economic pipe diameters and the length of pipe were determined. Agitators and pumps were also suitably designed. Appropriate motorized valves, timers, and control devices were chosen. The building size was chosen for the most compact size consistent with replacement and maintenance of equipment. The detailed costs of these items are given in Table C-I.

3. The annual acid costs were calculated directly from the effective flowrate of the elutant, the yearly operating period, and the unit cost of the acid. These and the equipment costs were summarized in Table III.

4. The costs for the other fixed bed designs were determined for all factors except labor and acid by using the 0.6 power of the size ratio, based on the volume of resin. The labor cost was a constant and the acid cost was calculated, as above, for each specific design.

Removal of a Trace Ion

The design of a fixed bed for ion removal is quite similar to that for ion separation, as can be seen by comparing the two diagrams in Figure C-1. In order to compare the two processes, the amount of B in the product solution or treated solution was taken to be the same for both cases, i.e. Area II was the same fraction of the area under the ion B curves. The ion B curves on Figure C-2 can thus be used for the calculations.

One other assumption is necessary before the designs can be undertaken. As pointed out before, the feed solution becomes the treated solution after the majority of ion B is removed from it, so the ratio of their quantities, t_1/T_2 , must be unity.

At $t_1/T = 1$ there are a large number of combinations of T_B and s_B that can be chosen. Also, as before, values of tp_2 can be calculated from s_B , so

at $t_1/T = 1$ and R_B and 3.68%

s_B	400	100	25	13	10
T_B	408	95	19	8.0	5.2
t_{B2}	886	235	66	38	30
$\frac{t_{B2}-T_B}{T_B}$	1.17	1.48	2.48	3.74	4.85

This information is converted into resin and elutant requirements and into costs in the same manner as outlined for the separation of trace components. The optimum combination leads to the costs indicated in Table IV.

Removal of a Gross Ion

The design of a fixed bed for gross ion removal requires the evaluation of an even more complex concentration history relation; see Hiester and Vermeulen (7), Equations (1) and (4). The amount of calculation required would have been excessive, so an approximate concentration history curve was constructed as shown in Figure C-3. The first curve covering area II and IV was calculated from

$$\frac{x_B}{(x_B)_0} = \frac{1}{1 + e^{(r-1)(t-s)}} \quad (8-30)$$

which is good for low r ($\approx \frac{1}{K_{BG}}$) values. The elution curve covering area VI is also good for low r values:

$$\frac{x_B}{(x_B)_0} = \frac{\sqrt{r-r} \sqrt{t'/s'}}{\sqrt{t'/s'} - r \sqrt{t'/s'}} \quad (8-31')$$

where $t' = t - t_E$

and where $\frac{s'}{s} = \left(\frac{h}{s}\right)_{\text{feed}} / \left(\frac{h}{s}\right)_{\text{elutant}}$

as indicated by evaluation of the correlation, Equation (4-34').

Area V is rectangular and its width is chosen to make the total area under the curve equal to T . As before

$$R_B = \frac{\text{Area II}}{T}$$

and the point t_1 was chosen by trial and error until this requirement was satisfied. The position of t_3 was chosen as the point at which $x_B/(x_B)_0 = 0.995$ and the position of t_2 as the place where $x_B/(x_B)_0 = 0.005$. Graphical designs of this type were prepared for $s = 13, 25, 100$, and 400 . The results obtained are as follows:

at $t_1/T = 1$ and $R_B = 3.68\%$

s_B	400	100	25	13	3
T_B	415	104	25	11.4	0
t_{B2}	955	238	58.4	28.8	
$\frac{t_{B2}-T_B}{T_B}$	0.38	0.38	0.40	0.44	∞

The fixed bed transfer units were converted to resin volumes by the method of Vermeulen et al (11), using their transfer height correlation. The optimum cost combination was obtained in the same manner as before and led to the costs indicated in Table IV.

TABLE C-I

47

CAPITAL EQUIPMENT COSTS FOR FIXED BED
SEPARATION PLANT

70% Recovery, 95% Purity of A in Product

<u>Item</u>	<u>Size</u>	<u>Material</u>	<u>No. Reqd.</u>	<u>Unit Cost</u>	<u>Total Cost</u>
Equipment					
Columns	2'D x 4'	glass lined	2	\$ 650	\$ 1,300
Surge Tanks	4'D x 3'6"	304 stainless	2	1,100	2,200
Blending Tank	4'D x 4'9"	" "	1	1,400	1,400
Blending Tank	4'D x 5'3"	" "	2	1,500	3,000
Surge Tank	4'D x 6'	" "	1	1,300	1,300
Surge Tank	4'D x 7'9"	" "	1	2,000	2,000
Centrifugal Pumps		" "	5	350	1,750
Diaphragm Pump		glass lined	1	1,000	1,000
Misc. Accessory Equipment					<u>3,600</u>
					\$17,550
Piping and Instrumentation					
Pipe and Fittings		304 stainless			2,600
Motor Gate Valves	1 1/2"	" "	13	120	1,600
Motor 3-way Valves	1 1/2"	" "	4	160	650
Check Valves	1 1/2"	" "	2	50	100
Flow Control Valves	1"	" "	2	200	400
Flow Controllers			2	600	1,200
Sight Glasses			2	75	150
Cycle Timer			1	500	500
Photometer Controller			1	6,000	<u>6,000</u>
					\$13,200

FIG. C-1
CONCENTRATION HISTORIES FOR TRACE ION SEPARATION AND REMOVAL
A-337-TR7-106

49

FIG. C-2
RELATIONS AMONG FIXED BED OPERATIONAL PARAMETERS

A-337-TR7-104

50

FIG. C-3
CONCENTRATION HISTORY FOR GROSS ION REMOVAL

A-337-TR7-108

7355 56

NOMENCLATURE FOR APPENDICES A, B, AND C

A, B, G = Ionic components A, B, and G, G being the eluting ion.

c_T = Total ionic level in the solution phase in meq/cc., equal to the sum of the ionic concentrations of components A, B, and G.

d_p = Mean diameter of resin particles, cm.

D = Distribution or partition parameter, dimensionless.

D_F = Effective counter-diffusivity of designated ion (with the eluting ion) in the solution phase, sq.cm/sec.

D_p = Effective counter-diffusivity of designated ion (with the eluting ion) in the resin phase, sq. cm/sec.

E = Ratio of the slope of the equilibrium line to the slope of the operating line, dimensionless.

E = Volumetric flowrate of elutant solution, cc/sec.

f_E = External void fraction of resin bed, dimensionless.

h = Height of resin bed, cm.

H_t = Height of transfer unit, cm.

I_0 = Bessel function of zero order, dimensionless.

J = Mathematical function based upon the integral of I_0 and defined in Equation (10-19b).

K = Chemical equilibrium constant for exchange, dimensionless.

M = Trace distribution coefficient, dimensionless.

N_C = Number of equilibrium contacts, dimensionless.

N_S = Number of equilibrium stages, dimensionless, $N_S - N_C/2$

$(Nt)_{CF}$ = Number of transfer units based on fluid film, dimensionless.

p = Purity or the fraction of a given component in a specific stream with respect to the amount of all components in that stream except the eluting ion.

Q = Total ionic concentration of the resin or the ultimate capacity, meq./g. oven-dried resin.

r = Equilibrium parameter, dimensionless.

- $\tilde{R}$ = Recovery or the fraction of a given component in a specific stream with respect to the amount in the incoming feed stream over the same time interval.
- R = Volumetric solution flowrate through fixed bed, cc/sec.
- R_F = Volumetric solution flowrate in moving bed, cc/sec.
- R_P = Volumetric resin flowrate in moving bed, cc true resin volume/sec.
- Re = Reynolds number, dimensionless.
- s = Number of fixed bed transfer units for saturation, dimensionless.
- s^0 = Number of fixed bed transfer units for elution, dimensionless.
- S = Cross-sectional area of column, sq. cm.
- Sc = Schmidt number, dimensionless.
- t = Solution capacity parameter, dimensionless.
- t^0 = Difference between t and t_E , dimensionless (see Figure C-3).
- t_1 = Solution capacity parameter denoting the time at which the effluent is diverted from treated solution storage to waste storage, dimensionless.
- t_2 = Solution capacity parameter denoting the end of the fixed bed cycle at the point where the resin is eluted to the desired degree, dimensionless.
- t_E = Solution capacity parameter denoting the point at which x/x_0 becomes 0.995, dimensionless (see Figure C-3).
- t_S = Solution capacity parameter denoting the end of the period at which x/x_0 ceases to be 0.995, dimensionless (see Figure C-3).
- T = Solution capacity parameter for the saturation period, dimensionless.
- u, v = Variables.
- V_F = Volume of solution aliquot introduced to the equilibrium-stage contactor.
- W_P = Weight of resin aliquot introduced to the equilibrium-stage contactor.
- x = Equivalent fraction of a component in solution with respect to the total ionic level of the solution, dimensionless.

- x_0 = Equivalent fraction of a component in the feed solution, dimensionless.
 x^* = Equivalent fraction in the solution in equilibrium with a given equivalent fraction on the resin, dimensionless.
 y = Equivalent fraction of a component on the resin with respect to the total capacity of the resin, dimensionless.
 y_0 = Equivalent fraction of a component on the entering resin, dimensionless.
 γ = Degree of approach to equilibrium transfer for a component in the solution phase, dimensionless.
 δ = Separation factor between B and A.
 ρ = Density of solution, g/cc.
 ρ_p = Density of resin, g oven-dried resin per cc of swollen volume.
 ξ = A variable associated with height or with column capacity.
 τ = Time, sec.

Subscripts

A, B, G = Components A, B, and G.

elut = Elution section

entr = Entering composition

exit = Exiting composition

sat. = Saturation section

Notes: The symbols underlined with a horizontal, wavy line in the text are equivalent to bold face symbols in the figures, i.e. E = **E**.

I - I

1 - 53

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