
# TECHNOLOGY OF PULSED SIEVE-PLATE EXTRACTION COLUMNS

## PART I — CARTRIDGE DESIGN.
## PART II — INTERFACE LEVEL CONTROL.

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## RESUMO

O comportamento de uma coluna pulsada equipada com diferentes conjuntos de placas, na extração do $HNO_3$ de uma solução 3N com TBP a 45 v% em Varsol, foi estudado. Os resultados foram comparados com aquêles obtidos com os mesmos conjuntos contendo zonas intermediárias de decantação (ISZ). As zonas intermediárias de decantação, como estudadas, não melhoraram o comportamento da coluna, e causaram um deslocamento indesejável das curvas de HETS e capacidade em direção à zona de maior energia de pulsação. Entretanto, o conjunto de Lucite - mostrou um pequeno acréscimo tanto na capacidade como na eficiência.

O efeito de temperatura na capacidade de sufocamento dos conjuntos de aço inoxidável, Lucite e mistos foi também estudado. Curvas de sufocamento determinadas a 22, 25 e $30^{\circ}$ C mostraram um aumento apreciável da capacidade com aumento da temperatura no caso dos conjuntos de aço inoxidável e Lucite. Dêste modo a temperatura mostrou-se como um importante parâmetro operacional.

O comportamento de um controlador de nível de interface, operando pelo princípio da diferença de condutividades elétricas dos líquidos, foi discutido na 2ª Parte. O controlador operou durante 80 horas em serviço normal e parece depender largamente de eletrodos bem projetados para operação satisfatória.



## RESUMÉ

On a étudié le fonctionnement d'une colonne pulsée, garnie des différents ensembles de plaques perforées pendant l'extraction d'acide nitrique 3N avec une solution

45 v% de tributylphosphate et 55 v% de Varsol. Les résultats ont été comparés avec ceux obtenus en présence des zones intermédiaires de décantation. Les zones intermédiaires de décantation produisent en général un déplacement indésirable des courbes de HETS et de l'engorgement vers la zone des hautes énergies de pulsation, sans améliorer la performance de la colonne. Dans le cas de l'ensemble en Lucite on a observé cependant une legère augmentation de la capacité et de l'efficacité de la colonne.

On a étudié aussi l'effet de la température sur la capacité de la colonne. Les courbes de l'engorgement en fonction de la température entre 22 e 30° C montrent une augmentation appréciable de la capacité avec la température croissante dans le cas des ensembles en Lucite et en acier inoxydable. La température est sans doute un important facteur operationel.

Dans la seconde partie du rapport, on présente des résultats préliminaires sur le fonctionnement d'un système automatique de réglage d'interface basé sur un détecteur conductométrique. Le système a fonctionné pedant 80 heurs, mais exige une mise au point additionnelle des électrodes.

## ABSTRACT

The performance of several cartridges was studied in the extraction of $HNO_3$ from a 3N aqueous solution into 45 v% TBP-in-Varsol. Results were compared with the ones obtained with the same cartridges containing intermediate settling zones (ISZ). Intermediate settling zones, as studied, did not improve column performance as expected, and caused an undesirable shifting of both HETS and flooding capacity curves toward the high energy region. The Lucite cartridge did show a small increase both in capacity and efficiency.

The effect of temperature on the flooding capacity of the stainless steel, Lucite and mixed cartridges was also studied. Flooding curves were determined at 22, 25 and 30° C, and show an appreciable increase in capacity with increasing temperature in the case of the stainless steel and Lucite cartridges. Thus, temperature proved to be an important operating parameter.

The performance of a conductivity-probe interface level controller is discussed in Part II. The controller operated for 80 hours in actual service and seems to depend to a large extent on proper electrode design for reliable operation.

## ACKNOWLEDGMENTS

The authors are indebted to Miss A. Pellegrini and Mr. O. W. Martins of the Electronics Division for the design of the level controller and to Mr. S. C. Caralt for carrying out most of the experimental work. Further thanks are due to the SERAC staff members of the Fontenay-aux-Roses C.E.A. Research Center, who suggested to one of us (K.J.Bril) the use of intermediate settling zones for improvement of cartridge performance. The pulse column employed in the present work was kindly lent by Orquima S.A.

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## 1. INTRODUCTION

Pulse columns, originally described by Van Dijck (19), have, since 1948 (6), found widespread use in nuclear separation processes.

Since nuclear applications require a high degree of efficiency, the technology of pulse columns has been extensively investigated and factors affecting column performance established by many workers (4, 5, 8, 9, 12, 14, 16, 17, 18). Despite the fact that the technology of pulse columns developed quickly, the understanding of the effect of column geometry, cartridge design and operating variables is still incomplete - and leaves room for much systematic work. In particular, the question of proper cartridge design is still a controversial matter. Whereas some authors consider cartridge design as a matter of great importance (11, 15), others simply rank it among second or third order factors (18).

In this work we have endeavoured to accumulate some - comparative data on different cartridges and improve column performance by introducing intermediate settling zones. Efficient intermediate settling zones should bear upon column performance, as they would eliminate the tight dispersion zone found in the middle of the column. The fact that the tight dispersion zone had an adverse effect on column performance has been previously pointed out (7, 11, 12, 13) and led to the development of the graded cartridge (12).

Along with the study on cartridge performance, it is also presented in Part II of this report, some preliminary results on the operation of a conductivity-probe level controller. Many types of level controllers have been described in the literature (Cf. Part II, p. 27), and these operate with different interface detecting devices, e.g., air-purge-type dip

tubes, capacitance probes, resistivity probes, floats, etc. Among all these detecting devices, conductivity probes seem to be the most suitable, at least for small solvent-extraction - columns.

## 2. PART I - CARTRIDGE DESIGN

**Foreword.** Factors affecting pulse column behavior can be divided into two main groups: design factors and operating factors. Plate spacing, plate hole diameter, hole distribution, percent plate free area, plate material, contacting section height and column diameter, are among the design factors. Operating factors are those which may be varied for a column of fixed design, such as amplitude, frequency, pulse-wave shape, flow rates, choice of continuous - phase and temperature. The effect of these factors has been described by many authors (4, 5, 7, 8, 12, 14, 16, 17, 18) and much work has been done in trying to correlate them to column performance.

The subject of cartridge design, which comprises the group of factors such as material of plates, plate spacing, - plate hole diameter and percent free area is the main objective of this work. The performance of several different cartridges has been studied and results compared with those obtained using the same cartridges, but with intermediate settling zones.

Experiments were conducted at organic phase continuous, constant pulse frequency, constant flow ratio and variable pulse amplitude. The efficiency of the column was studied at constant total flow. Flooding curves were determined at constant temperature.

The extraction of $HNO_3$ from a 3N aqueous solution into 45 v% TBP-in-Varsol has been chosen for this study but the work was undertaken to serve as a model for optimization studies of cartridge design for the I.E.A. uranium and thorium purification plant.

## EXPERIMENTAL

Description of Apparatus. A schematic diagram of the experimental equipment is shown in Fig. 1. The pulse column consists of 1) a contact zone, containing the perforated plate assembly, 2) top and bottom disengaging sections, to allow enough time for emulsion breaking, 3) pulse generator and transmission line.

The contact zone is composed of a 5.6 cm-I.D. Pirex glass tube, 1.20 m high, containing 23 tight-fitting perforated plates, positioned in place by tubular 5 cm-long Lucite spacers which slide along the center rod. Top and bottom disengaging sections consisted of cylindrical glass reservoirs, approximately 2.5 liter capacity, soldered to the column proper. Inlet and outlet connections were as shown in Fig. 1.

The pulse generator was a home-built type unit, consisting of a 1/3 Hp, 1730 rpm electric motor, different-sized pulleys and belts, and one 1/13 speed reducer, for pulse frequency adjustments; a flat-faced disk placed at the low-speed shaft of the speed reducer, provided with holes positioned at different radii, permitted adjustment of the length of the piston stroke. Piston packing was Teflon-impregnated asbestos. Piston and cylinder were both built of AISI 316 stainless steel. The assembly is assumed to produce a near-sinusoidal pulse wave.

Process liquids were pumped to overhead tanks and flowed to the column by the effect of gravity [(a)], after passing through Lucite rotameters. A constant-temperature water-bath adjusted the temperature of the incoming organic phase before entering the rotameter. Outgoing streams were collected in calibrated glass con-

(a) Because of the limitation imposed on available height, the organic phase flow rate could not be increased over 80 l/hr.

tainers. The organic phase level in the overhead tank was maintained constant within $\pm$ 10 cm by means of a level switch which actuated the organic-phase pump motor.

The interface level inside the column could be maintained constant either by controlling the position of the aqueous phase outlet, or by means of an interface level controller (See Part II).

Different types of plates were used in this study, namely stainless steel plates, plastic and nozzle-plates. Their characteristics were as shown in Table 1.

Table 1. Characteristics of Plates. Typical values.

| | Plastic No. 1 | Plastic No. 2 | Stainless Steel | Nozzle-Plate |
|---|---|---|---|---|
| Material of Plate | Lucite | Lucite | AISI 316 | AISI 304 |
| Thickness of Plate | 3.3 mm | 3.4 mm | 1.65 mm | 1.7 mm |
| Diameter of Plate | 55.1 mm | 54.5 mm | 54.8 mm | 54.4 mm |
| Diameter of Holes | 2.8 mm | 3.2 mm | 2.8 mm | 2.9 mm |
| Number of Holes | 78 | 66 | 78 | 28 |
| Percent Free Area | 20.3 % | 23.1 % | 20.4 % | 8 % |
| Hole Geometry | hexag. | hexag. | hexag. | rectang. |
| Nozzle Depth | 0 | 0 | 0 | 2.0 mm |

The mixed cartridge consisted of 11 No. 1-Lucite plates and 12 stainless steel plates, displaced 5 cm apart from each other, in alternate pairs.

Reagents. Technical grade TBP used in the experiments was purified by the usual procedure (3,4). Varsol, a paraffinic naphta fraction having a density of 0.790 g/cm$^3$ at 15.5° C and a boiling range of 160 to 198° C, was used as the diluent. Commercial grade Varsol is produced by the Esso Standard of Brazil and is purified before use to remove unstable aromatic and olefinic hydrocarbons (3). The treatment consisted of heating for 6 hours at 75° C with 10 v% con-

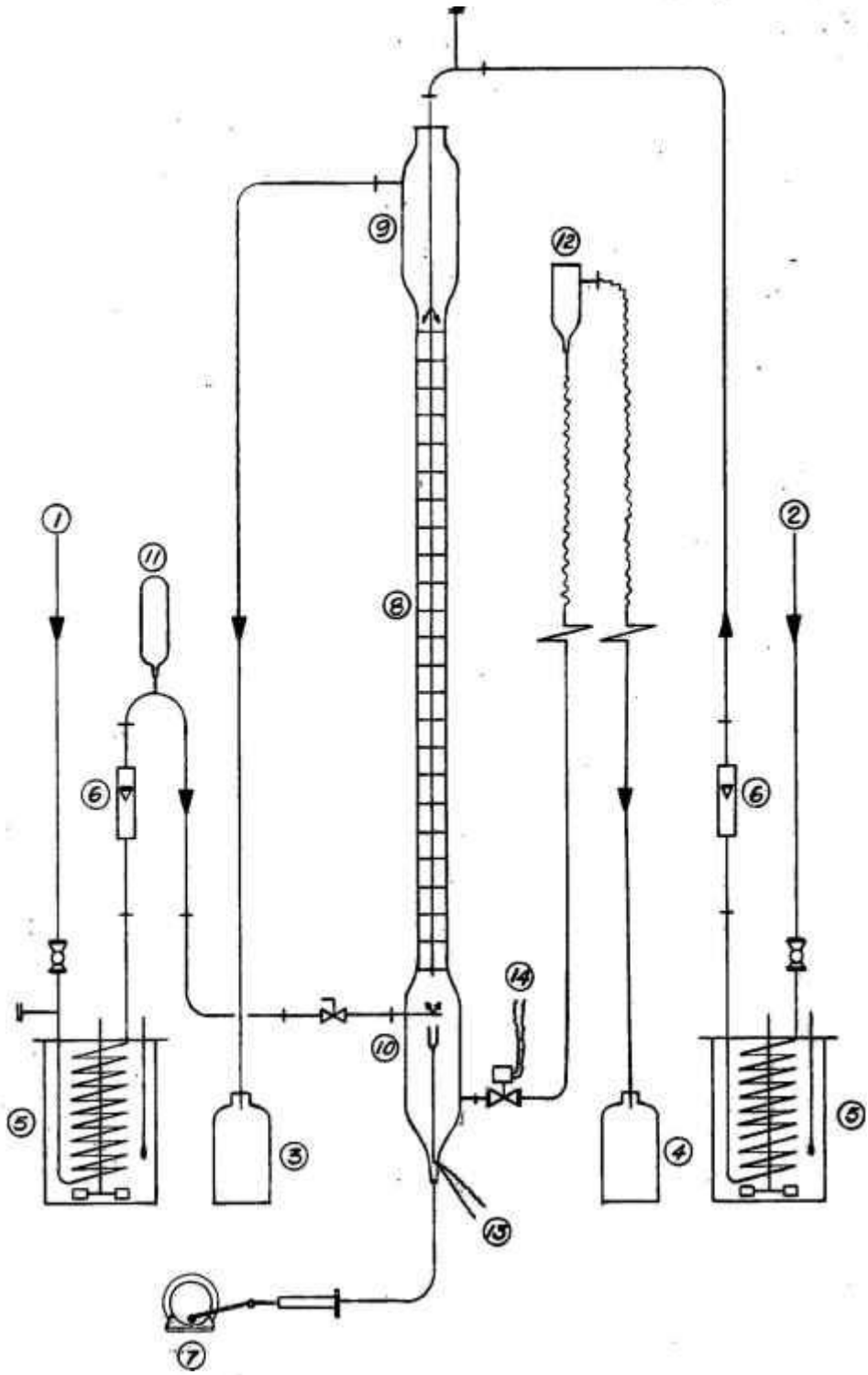



Fig. 1 - Schematic Diagram of Experimental Equipment.

KEY TO FIGURE 1:

1 organic feed stream from overhead tank

2 aqueous feed stream from overhead tank

3 extract collecting bottle

4 raffinate collecting bottle

5 thermostatic water-bath

6 rotameters

7 pulse generator

8 contacting section

9 top disengaging section

10 bottom disengaging section

11 surge bottle

12 aqueous overflow

13 electrode wires to controller

14 solenoid wires from controller

centrated sulfuric acid, followed by washings with dilute sodium carbonate solution and acidified water.

Technical grade concentrated nitric acid ($42^{\circ}$ Bé), was used for preparing the 3N aqueous solutions. Water used for these solutions was plain tap water.

Experimental Conditions. Experiments were conducted at organic phase continuous, constant flow ratio, constant pulse frequency and variable pulse amplitude. HETS values were determined at constant total flow and at room temperature, which varied between 22 and $28^{\circ}$ C. Temperature was held at $25^{\circ}$ C for the flooding determinations. Experimental data are presented in Tables A. 2.1 to A. 2.4, Appendix A.

The organic phase consisted of tributyl-phosphate diluted with Varsol to 45 volume percent. The TBP concentration was kept at $45 \pm 0.2$ v%. The aqueous phase was a $3 \pm 0,02$ N nitric acid solution.

HETS values were determined at 20 and 2 liters per hour respectively, for the organic and aqueous incoming-feed flow rates (See App. A. 2.1). This ratio was kept reasonably constant as shown in Tables B. 2.1 to B. 2.4.

All experiments were performed at a frequency of 102.8 cycles per minute, while the amplitude in the column was varied from 1.5 to 3.5 cm for the different experiments.

Intermediate settling zones were introduced by removing a total of six plates in the middle of the contacting section. The settling zone length measured approximately 35 cm, from plate-to-plate, in each case.

Operation of the Column. Several steps were followed

prior to setting the column into operation, namely, 1) adjustment of the water-bath temperature, 2) adjustment of the amplitude and frequency to the proper values, 3) filling the bottom reservoir of the column with water up to a certain level, 4) filling the remaining of the column with the organic phase, 5) checking the operation of the pulser.

After the previous steps were done, the pulser was started and the rotameters adjusted to the desired reading. Outgoing liquid streams were collected into calibrated flasks with a bottom outlet provided with a stopcock, and then allowed to flow into larger containers. When a check reading was desired on the flow rate of either phase, the stopcock was closed and a certain volume timed.

HETS Determinations. After a three-hour period of operation, samples were drawn from both outflowing phases and analized for their acid contents. From there on, every half hour samples were analized until steady state operation was attained. Steady state operation was assumed to take place when after a one-hour period no change in concentration of both phases was found. At this point the densities of the leaving streams were taken at 25° C, and the flow into the column stopped. Operation was over. Each experiment lasted an average of 6 hours.

The acid contents of the ingoing liquids were analized prior to beginning the operation, and their densities determined at 25° C. The TBP content of the organic phase was analized and adjusted after a set of three experiments.

Flooding Determinations. Since experiments were carried out at organic phase continuous, flooding was assumed to occur when enough aqueous phase accumulated above the top sie

plate and started to go up slowly towards the organic phase - outlet. The ingoing stream flows of both phases at this point were considered as the flooding rates at the existing conditions. When determining flooding rates, the aqueous and organic flows were increased stepwise, keeping a constant flow ratio between the phases, until flooding was attained. Flooding occurred within 10 to 15 minutes after the last step change in entering flows. As temperature affects flooding rates (Figs. 11, 12, 13), all flooding determinations were done at constant temperature.

Analytical Methods. TBP concentration in Varsol was determined by the acid saturation method (1, 2), with an accuracy of 1%.

Nitric acid concentration in the organic phase was determined by titration with 0.1 N sodium hydroxide solution, by means of a piston-buret. 25 ml of the organic phase were measured and 100 ml of deionized water added to it. The mixture was titrated under agitation by using bromthymol blue as indicator.

The nitric acid content of the aqueous phase was analized by titration with 0.1 N sodium hydroxide solution in a piston-buret, using methyl red as indicator.

The x-y Diagram. Fig. 2 shows the x-y diagram for the $HNO_3$-Water-45 v% TBP-Varsol system. Fig. 3 shows the distribution coefficient of $HNO_3$ versus the aqueous phase concentration for the same system. Data for these plots are given in Appendix A, Tables A. 1.1. and A. 1.2. The equilibrium points were determined at three different temperatures, i.e., 25, 30 and 35° C. Results show that in this temperature range the effect of temperature on the distribution coefficient of $HNO_3$ is neg-

ligible, therefore a single line was drawn through the experimental points.

Fig. 2 was used for carrying out the graphical calculations of the number of ideal extraction stages by the McCabe-Thiele method. During the actual calculations, the low portion of the graph was expanded into another graph paper (Cf. Fig. C.1, p. 45) to permit a better approximation of stage fractions. All points were calculated from the same graphs. The choice of the coordinates, grams of $HNO_3$ per gram of $HNO_3$-free solvent, results in a straight operating line independently of volume changes of $HNO_3$ along the column, which increases the organic flow and decreases the aqueous flow (10).

Calculation of HETS's. The calculation of the height equivalent to a theoretical stage (HETS) was done in the usual manner, i.e., calculation of the number of theoretical stages by the stepping procedure of McCabe-Thiele and division of the extracting section height by the number of stages found. The operation line was drawn in on Fig. 2 by using the nitric acid concentrations of the ingoing and outgoing streams at steady state operation of the column (Cf. App. C).

Comparison of Cartridge Performances. The choice of an adequate cartridge for a certain operation is primarily an economic consideration striving to find the lowest af that will result in a high throughput without impairing the necessary efficiency. In general one is faced with one of the two possibilities, viz., choosing the cartridge before construction of the column or choosing the cartridge for a column already - built. Nevertheless, while in the former case there is greater latitude in the choice both situations lead to finding which cartridge gives the highest capacity for a value of HETS within a certain range.

For comparison of cartridge performances, plots of HETS and flooding capacity versus af are prepared (Figs. 4, 5, 6, 7, 8). For an actual designing problem where the allowable range of HETS's is known, the best cartridge is the one offering the highest througput in this range, and it is readily found from the previous plots.

When the HETS range is not defined, both HETS and flooding capacity must be taken into consideration. This can be done by introducing a new parameter, arbitrarily named performance factor ($P_f$), and defined as the ratio of capacity to HETS. As will be shown in a forthcoming report, this ratio is directly related to production capacity of the column. A plot of $P_f$ versus af is shown in Fig. 10. (Cf. footnote on page 15).

## RESULTS

Presentation of the Data. Data are presented as plots of HETS and flooding capacity versus af for each different type cartridge (Figs. 4, 5, 6, 7, 8). In the same plots is also shown the effect of one 35 cm-intermediate settling zone (ISZ) on HETS and capacity. Data pertaining to these plots are found in Appendix B.

Reproducibility of Data. From a total of 42 runs of HETS for the different cartridges, 39 points are reported as shown on the accompanying graphs and 3 points discarded as resulting from blunt errors. From the 39 points reported, three were reruns which checked within 0%, 0% and 8% respectively, the latter being obviously a bad point (Cf. Fig. 4).

All flooding data obtained at 25° C are reported. The average relative error was 3.2% and the maximum 6.6%.

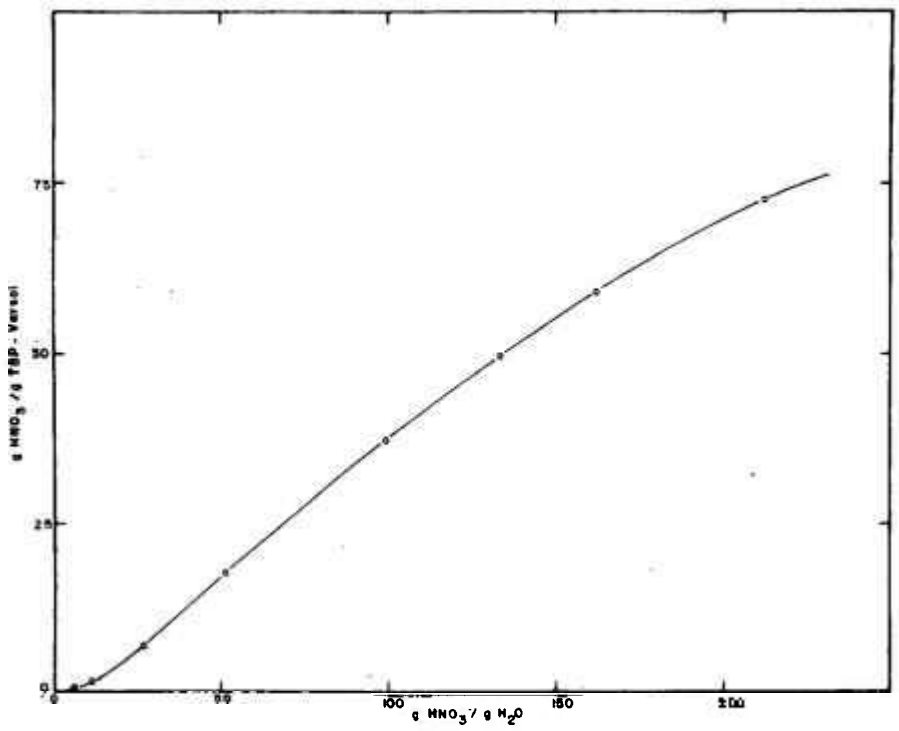
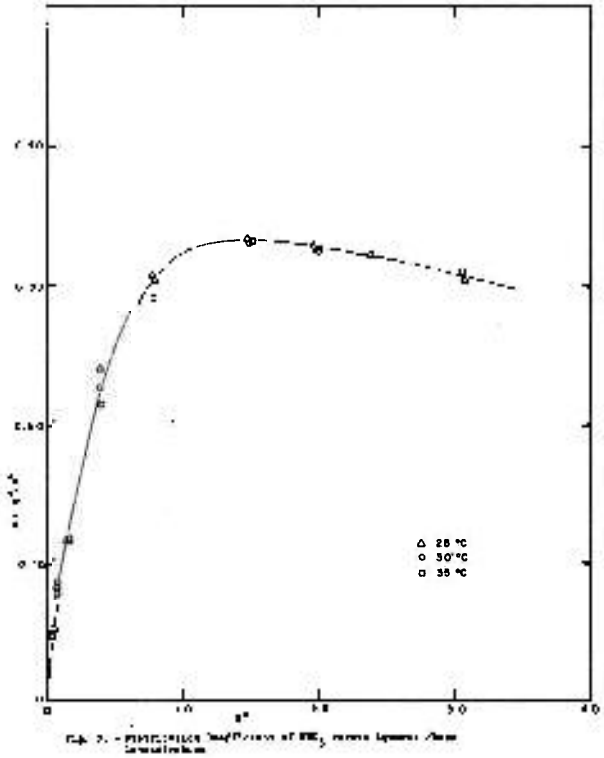



Fig. 2. - Equilibrium Diagram for the $HNO_3$-Water-45 v% TBP-Varsol System



Evaluation of Results. Evaluation of cartridge performance was done from $P_f$ values at the af's of maximum efficiency and at a point 10% lower than this. Results are shown in Tables 2 and 3, and in Fig. 9.

The reason for comparing performance factors at two different values of af, is because the flooding curve has a steep negative slope which is different for each cartridge, whereas the HETS curve can be rather insensitive to af fluctuations (e.g., stainless steel cartridge). Thus, one could find that a certain cartridge having poor capacity at its maximum efficiency af, could give satisfactory results at a lower af without appreciable increase in the HETS. This is actually the case of the complete mixed cartridge, as seen from Table 2 and 3, where a 10% change in af, increased the capacity by 42% while the HETS increased only by 4%.

Table 2. Performance Factors at Maximum Efficiency af's

| Cartridge | af (cm/sec) | capacity (l/cm2/hr) | HETS (cm) | $P_f$ ($hr^{-1}$) |
|---|---|---|---|---|
| Mixed Cartridge (complete) | 4.26 | 1.55 | 25 | 62 |
| Mixed Cartridge (with ISZ) | 4.82 | 1.55 | 25 | 62 |
| Stainless Steel (complete) | 4.46 | 1.8 | 25 | 72 |
| Stainless Steel (with ISZ) | 5.1 | 1.4 | 25 | 56 |
| No. 1-Lucite (complete) | 3.56 | 1.8 | 29 | 62 |
| No. 1-Lucite (with ISZ) | 4.62 | 1.4 | 27 | 52 |
| No. 2-Lucite (complete) | 4.1 | 1.6 | 26 | 62 |
| Nozzle-Plate (complete) | 2.9 | 2.1 | 36 | 58 |

Table 3. Performance Factors at an af 10% Lower than the Maximum Efficiency af.

| Cartridge | af (cm/sec) | capacity (l/cm2/hr) | HETS (cm) | $P_f$ ($hr^{-1}$) |
|---|---|---|---|---|
| Mixed Cartridge (complete) | 3.83 | 2.2 | 26 | 85 |
| Mixed Cartridge (with ISZ) | 4.34 | 1.8 | 26 | 69 |
| Stainless Steel (complete) | 4.01 | 2.4 | 26 | 92 |
| Stainless Steel (with ISZ) | 4.59 | 1.7 | 26 | 65 |
| No. 1-Lucite (complete) | 3.20 | 2.4 | 31 | 77 |
| No. 1-Lucite (with ISZ) | 4.16 | 1.8 | 29 | 62 |
| No. 2-Lucite (complete) | 3.69 | 2.1 | 31 | 68 |
| Nozzle-Plate (complete) | 2.61 | 2.9 | 38 | 76 |

From results presented above and the plots of $P_f$ vs. af (Fig. 9), one can easily conclude that the complete stainless steel cartridge is the best suited cartridge for the existing extraction conditions. Besides the fact that it shows the highest performance factors in both cases and its HETS ranks among the lowest found, its efficiency is almost unaffected by af changes within rather large limits, as shown by Fig. 5.

## DISCUSSION OF RESULTS

Performance of Cartridges. As pointed out in the previous section, the stainless steel cartridge gives the best compromise between high flooding capacity and high extracting efficiency.

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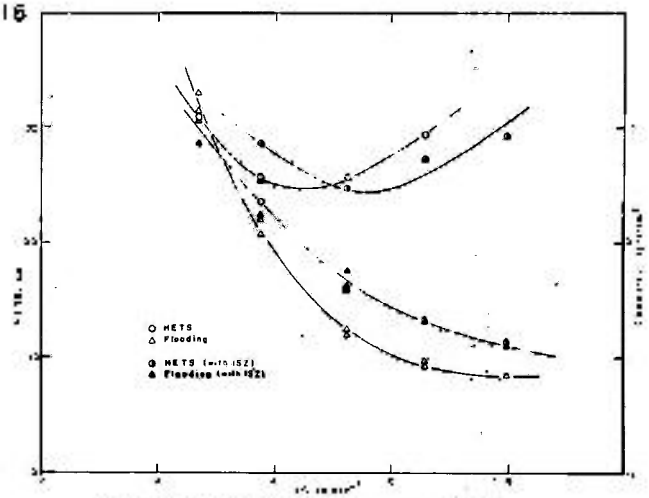
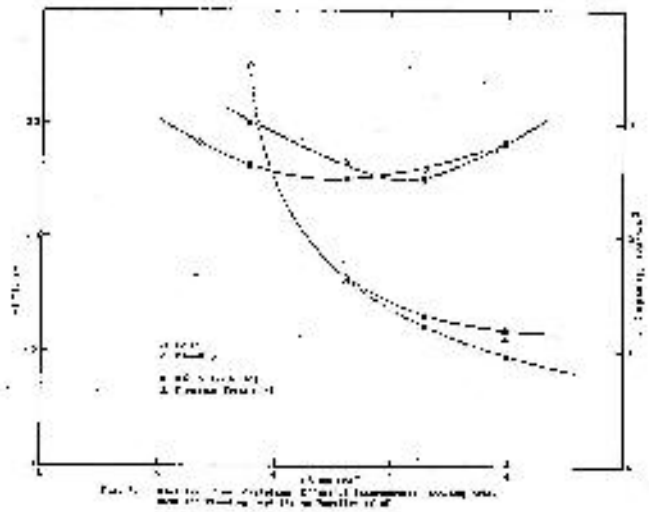
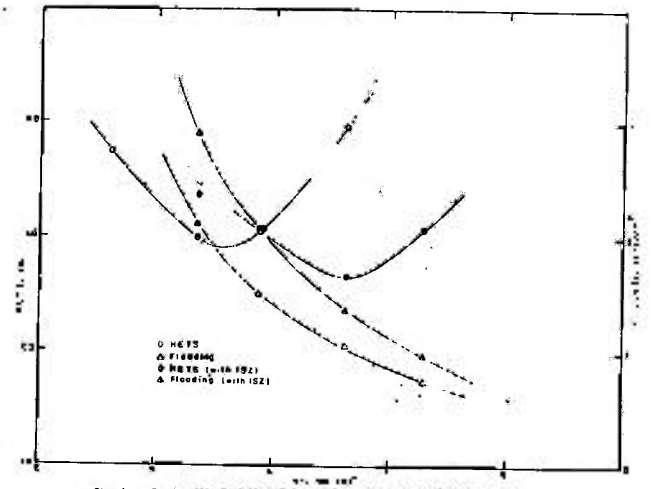
After completion of the present report we have found a reference given by Treybal, R.E. in "Chem. Eng. Progr." 5, p. 77 - (May 1964), to previous application of this factor which we arbitrarily named performance factor.


HETS
Flooding
HETS (with ISZ)
Flooding (with ISZ)




HETS
Flooding
HETS (with ISZ)
Flooding (with ISZ)

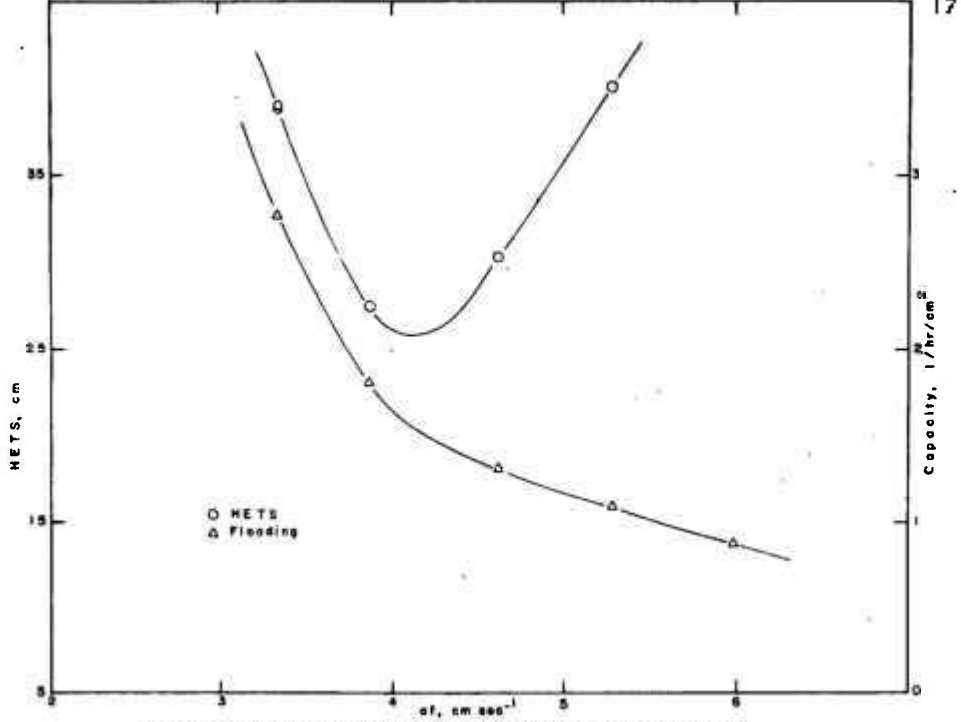



Fig. 7. - No. 2-Lucite Cartridge. HETS and Flooding Capacity as Function of $F_s$

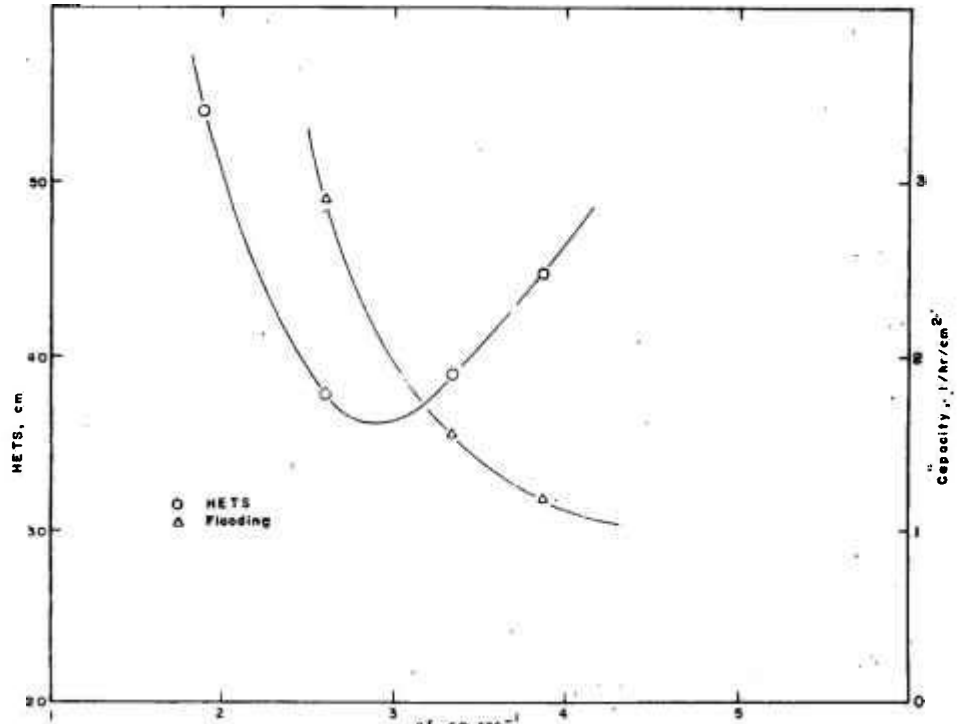



Fig. 8. - Nozzle-plate Cartridge. HETS and Flooding Capacity as Function of $F_s$.

The high efficiency of this cartridge can be explained as resulting from the cyclic coalescence and dispersion of the aqueous phase (dispersed phase), which results in a high rate of mass transfer, because there is a minimum of "aging" of the surface of the dispersed droplets. This phenomenon of the effect of coalescence on efficiency (11, 13), is well illustrated by the small sensitivity of the HETS curve to changes in amplitude. This cartridge being wet by the aqueous phase, coalesce the aqueous droplets, thus renewing constantly their exposed surface and maintaining an almost constant efficiency in a wide range of amplitudes.

The mixed cartridge used in this work performed well. HETS's found with this cartridge were among the lowest (Cf. Table 2). This cartridge consisted of alternate pairs of No. 1-Lucite and stainless steel plates and seemed to average, as expected, the properties of these two cartridges. Thus, one finds that its maximum efficiency af occurs near the average - of the maximum efficiency af's of these cartridges. The sensitivity of its HETS curve to af changes, is also intermediate between the low sensitivity of the stainless steel cartridge and the rather high sensitivity of the Lucite cartridge. Nevertheless, both its capacity and efficiency seem to follow the properties of the stainless steel cartridge closely.

The No. 2-Lucite cartridge introduced in this study in order to give an order-of-magnitude glimpse into the effect of larger diameter plate holes and larger clearance between column walls and plates, surprisingly enough, gave better HETS values than the No. 1-Lucite cartridge, though lower performance factors were obtained. The maximum efficiency af shifted towards higher values, in the same fashion as found with the introduction of intermediate settling zones in the other cartridges. From comparison between these effects one can infer

that whenever there is a decrease in the resistance to liquid flow through the column, due to removal of physical obstacles, larger amounts of energy must be supplied through the pulser to break up the liquid against the remaining obstacles in order to restore the same operating efficiency.

Nozzle-plates, which were also studied for comparing their efficiency under the existing conditions, operated poorly. This can be explained to a certain extent as resulting from the low aqueous to organic flow ratio, which hindered its performance by not forming jets of the coalesced aqueous phase, and thus betraying its original conception. As a consequence of its low plate free area, the capacity of the nozzle - plate cartridge at a given af is the lowest of all cartridges. Nozzles were oriented upward; one point was repeated with nozzles pointing downward (see Table A. 2.4) without any improvement (see Table B. 1.4).

Intermediate Settling Zones (ISZ). The introduction of settling zones had no striking effect on performance, contrarily to what was initially believed. Figs. 4, 5, show that in the case of the mixed and stainless steel cartridges there is only a shift of HETS and capacity curves towards higher af's without any substancial changes in either one. The only cartridge which did show an increase in efficiency was the No. 1- Lucite cartridge (Fig. 6); performance factors were also higher (Cf. Fig. 9), though shifting to higher af's occurred.

Effect of Temperature on Flooding Capacity. Figs. 11, 12 and 13, show the effect of temperature on flooding capacity for the different cartridges studied. As seen from Figs. 11 and 12, temperature effects were quite remarkable both in the No. 1- Lucite and stainless steel cartridges, while no appreciable effects were observed in the mixed cartridge capacity.

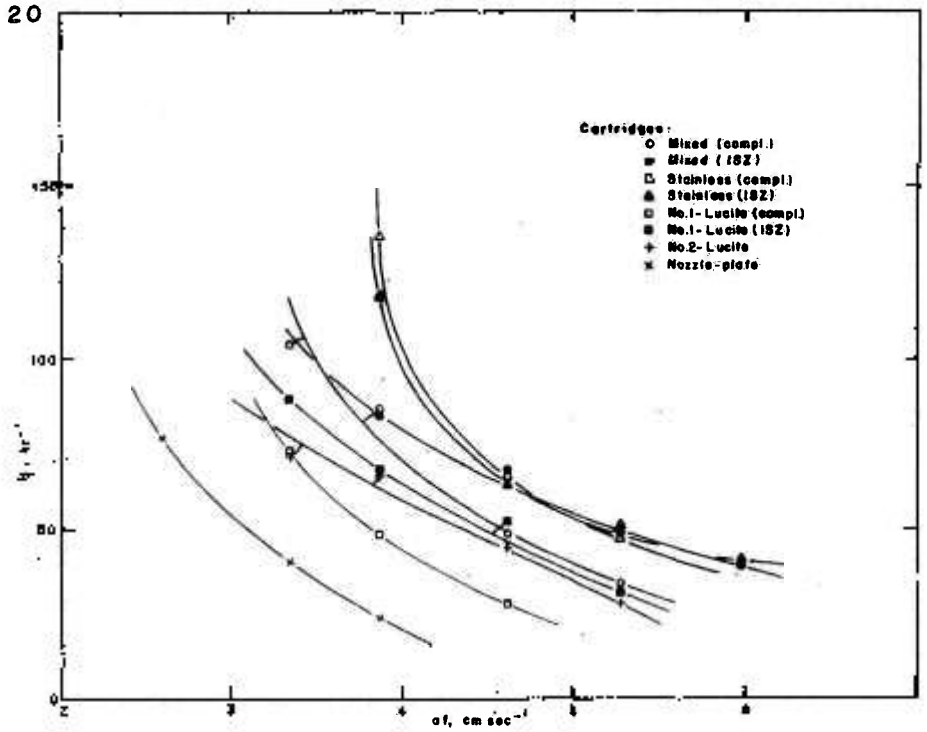
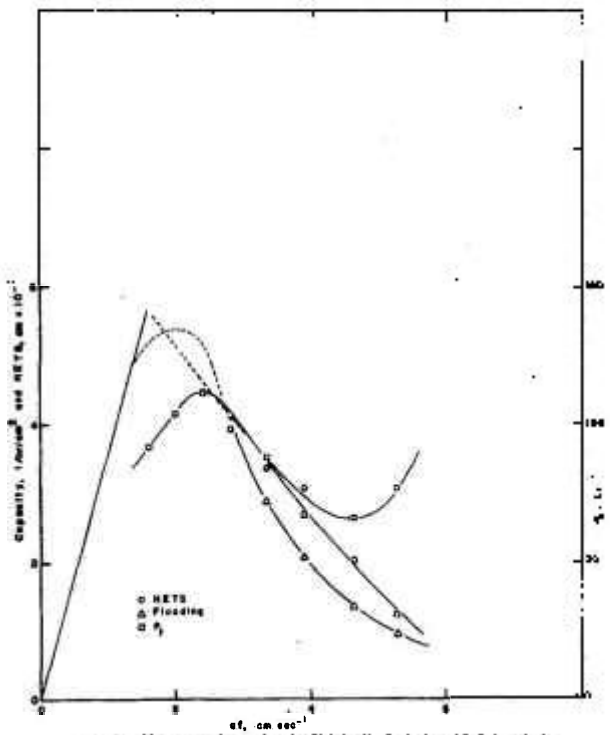



Fig. 9. - Performance Factors for the Different Cartridges as Function of af.



No reasonable explanation was found for this fact, and it is somewhat contradictory with expected results. Naturally one would assume that the temperature dependence for the mixed cartridge would lie somewhere between the effects observed in the two mother cartridges.

Results are quite significant as they give an order-of-magnitude idea of the effect of temperature on flooding, and serve to call attention to this effect which, despite being known, has been too often neglected in the past, even in attempts to generalized flooding correlations.

Conclusions. From analysis of the data, the following conclusions were drawn:

1) The performances of all cartridge designs studied were rather similar if compared at proper af's.
2) Intermediate settling zones, as studied, did not improve the performance of the mixed and stainless steel cartridges, and caused an undesirable shifting of both the capacity and efficiency curves toward the high amplitude region. A small increase in both capacity and efficiency of the No. 1-Lucite cartridge was observed.
3) In the series of nozzle-plates, No. 1-Lucite, No. 2-Lucite, mixed plates and stainless steel plates, the af values corresponding to optimum HETS's increase steadily from 2.9 to 4.46 cm/sec. Additional increase of optimum af is observed as a result of the introduction of the ISZ. Thus, one can infer that when there is a decrease in the resistance to liquid flow through the column by removal of physical obstacles (increasing hole diameter, clearance, free surface or decreasing coalescence efficiency), larger amounts of energy must be supplied through the pulser to break up the liquid and thus restore the same operating efficiency.
4) The best suited cartridge for the extraction conditions and

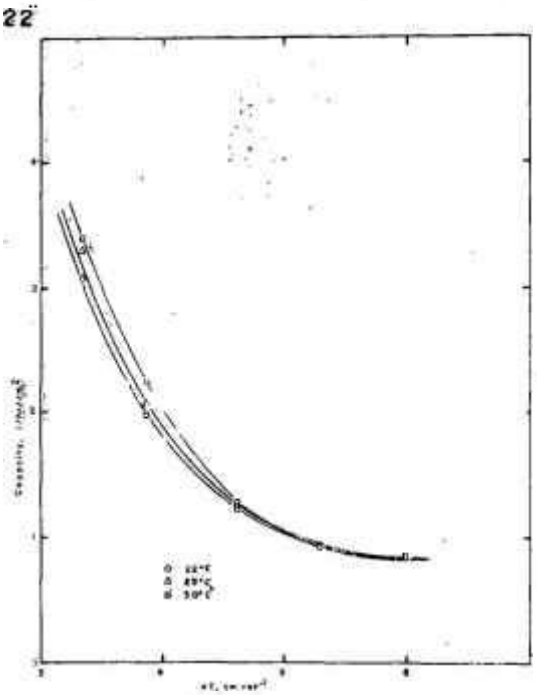


Fig. 11 - [illegible] Cartridge. Effect of Temperature on Flooding Capacity.

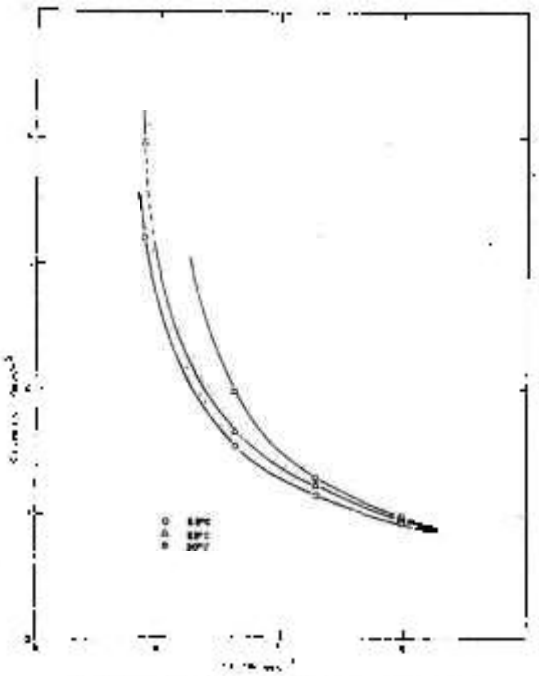


Fig. 11 - [illegible] Cartridge. Effect of Temperature on Flooding Capacity.

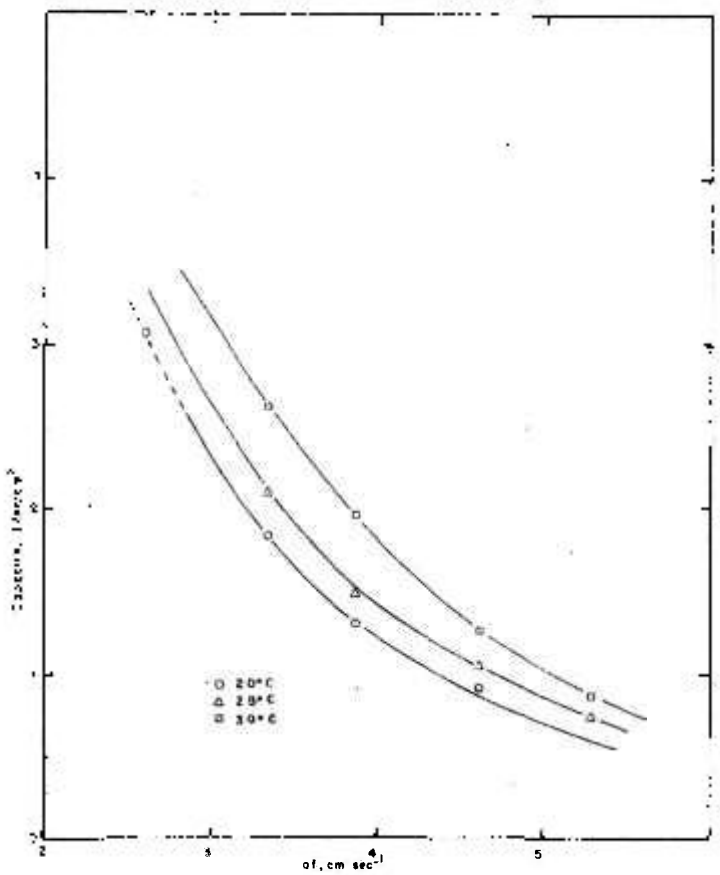



Fig. 13 - Dual-Lucite Cartridge. Effect of Temperature on Flooding Capacity.

system studied was the stainless steel cartridge. The efficiency of this cartridge is but little affected by rather large changes in af around the optimum value, so that the performance factor can be increased substantially without significant loss in efficiency.

5) The poor performance of the nozzle-plate is probably due to the low flow rate of the dispersed phase, which did not form jets through the plates, thus betraying its original conception.

6) Temperature is definitely an important operating variable and has not been given due attention in the past. The effect of temperature on flooding capacity of both the Lucite and stainless steel cartridges was quite important, while it did not show an appreciable effect on the mixed cartridge capacity.

Conclusions herein stated refer to the system and extraction conditions which prevailed during this study.

## 3. PART II - INTERFACE LEVEL CONTROL

Foreword. The interface level height in a pulse column is an important operating parameter, as it controls the residence time of the dispersed phase in the disengaging section. Therefore it is of the utmost importance that a constant level height be maintained to provide enough time for phase disengagement and insure a minimum of entrainment in the effluent streams.

The problem of level control resides mainly in that the interface level is a pulsating, emulsion-like region, which is not clear-cut and sometimes may appear as an emulsion continuum in passing from one phase to the other. Level-sensing mechanisms then, have to be so designed as to distinguish between a true interface or an emulsion, between a pulse pressure signal or a hydrostatic pressure variation, between the wave motion of the interface and its true vertical displacement, and also cope with all the dirt accumulated in the interface region.

Among the main methods of interface detection, we find: air-purge-type dip tubes, capacitance probes, conductivity probes and floats. Each of these methods have their drawbacks, and sometimes while performing well in pilot-plant scale, have been subject to frequent failures when applied industrially. Air-purge-type mechanisms for instance, can only be applied successfully in large installations, because they require constant flushing of the dip tubes with large volumes of water [(a)] and this may not be permissible in small-size plants.

Float-type mechanisms are far more expensive and complicated than either conductivity or capacitance probes, as their calibration and mounting are rather critical, and they

(a) Especially if the columns are operated with bottom interface level (organic phase continuous).

involve expensive accessories. The conductivity probe seems to be the most simple detecting device and it was decided to study its performance, in order to acquire enough experience to apply eventually this type of device in the control of industrial-scale columns.

## DESCRIPTION OF APPARATUS

The experimental equipment was mounted in conjunction with the column described in Part I of this report, and consisted essentially of platinum-tipped glass electrodes, an electronic detector and a solenoid valve placed in the aqueous effluent stream. The electrodes were introduced from the bottom, through the pulse leg, up to the height where the level was to be maintained (Fig. 1), with their tips leveled at the same horizontal plane.

The electronic circuit was as shown in Fig. 14. Whenever the electrodes were immersed in the low conductivity liquid, the relay was energized closing the solenoid valve. A capacitor was connected to the grid of the 12AU7 tube in order to provide a 17-second signal delay, thus enabling the controller to distinguish between a true interface movement and the constant cyclic input signal.

The solenoid valve was a normally-open-type valve, with a Lucite body, magnetic stainless steel plunger and Teflon seat.

## DISCUSSION

The level controller employed in this study operated satisfactorily during the limited amount of operating hours, which totaled approximately 80 hours of service.

The interface level could be maintained approximately equal to the pulse amplitude in the disengaging section, i.e.,

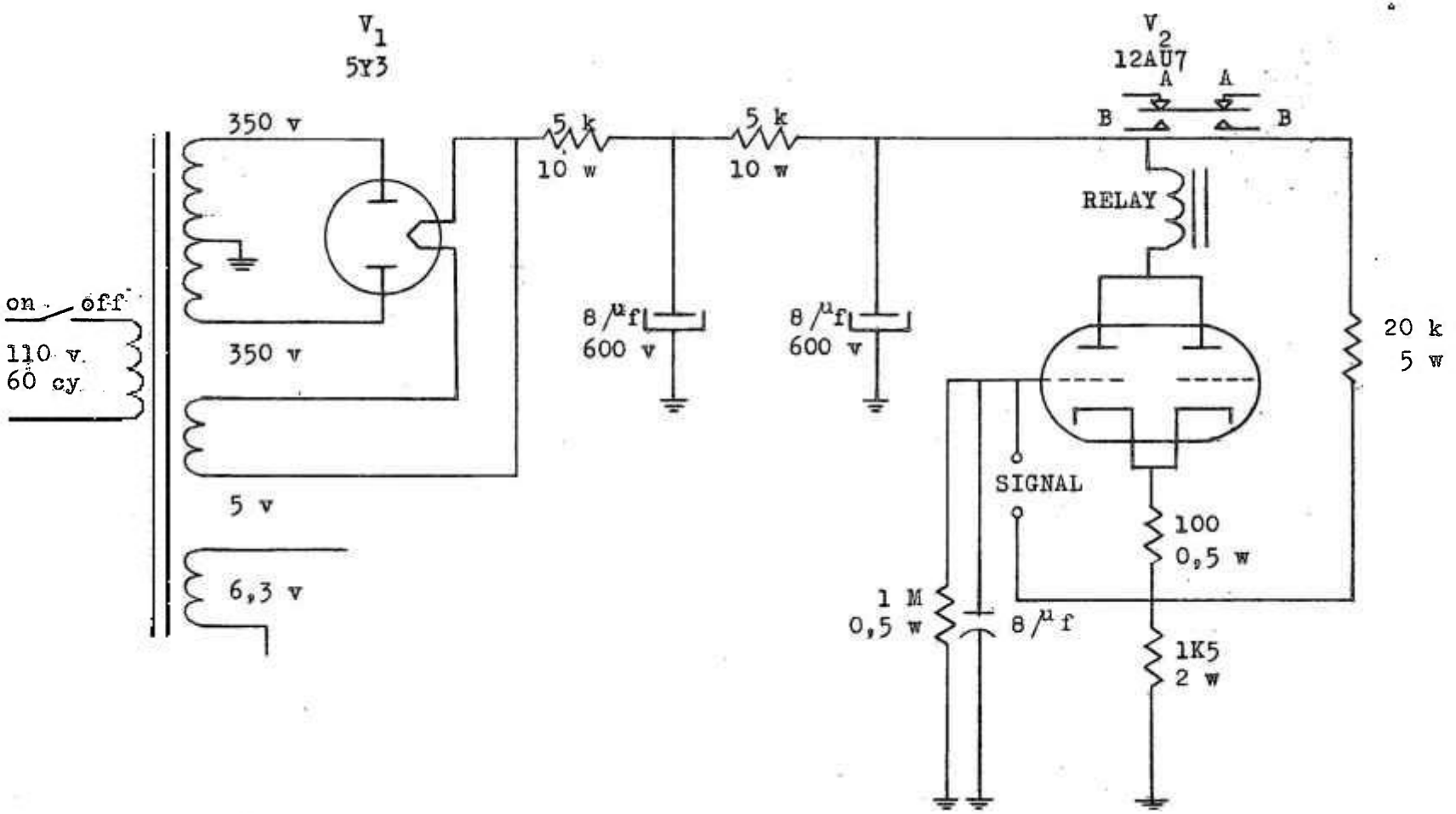



Fig. 14 - Interface Level Controller. Electronic Circuit.

0.5 to 1.5 cm. The response time lag of the controller being constant, for a fixed amplitude value, the increase in the control range over the amplitude is determined by the flow caused by the hydrostatic head between the aqueous outlet position and its equilibrium position. The equilibrium position is that which produces a flow rate identical to the incoming feed flow rate. In these experiments the aqueous outlet was maintained near the equilibrium position in order to keep the control range approximately equal to the amplitude.

The proper design of the detecting electrodes is of primary importance for successful operation of the controller. The original electrodes consisted of two platinum tips soldered to a glass bead, a distance of 1 cm apart from each other. This small distance caused sometimes short-circuiting of the electrodes, due to the aqueous-phase film which adhered to the glass surface. A new electrode design was then introduced to overcome this difficulty (Cf. Fig. 1), in an effort to avoid any major changes in the existing column. Electrodes to be used for the industrial columns will be mounted with their tips downward in two independent vertical entrances, electrically insulated from each other, to avoid surface currents.

Another possible source of trouble in the operation of these electrodes, might be scaling of their tips, which can prevent or delay the detection of the interface level. The problem is currently under study and it is envisaged the use of a constant vibration for cleaning the electrode tips.

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## 5. APPENDIX

A. Experimental Data

B. Calculated Data

C. Sample Calculation

## APPENDIX A.1 - EXPERIMENTAL DATA

Table A.1.1. - Distribution coefficient of $HNO_3$ - Organic Phase = 45.1 v% TBP-in-Varsol.

| t = 25° C | | t = 30° C | | t = 35° C | |
|---|---|---|---|---|---|
| K | $N^a$ | K | $N^a$ | K | $N^a$ |
| 0.052 | 0.048 | 0.050 | 0.048 | 0.047 | 0.049 |
| 0.080 | 0.09 | 0.083 | 0.093 | 0.0745 | 0.09 |
| 0.114 | 0.17 | 0.114 | 0.18 | 0.114 | 0.17 |
| 0.41 | 0.237 | 0.224 | 0.41 | 0.212 | 0.42 |
| 0.308 | 0.78 | 0.291 | 0.79 | 0.304 | 0.79 |
| 0.334 | 1.48 | 0.329 | 1.49 | 0.324 | 1.51 |
| 0.329 | 1.96 | 0.325 | 3.0 | 0.327 | 1.99 |
| 0.323 | 2.38 | - | - | - | - |
| 0.304 | 3.06 | 0.307 | 3.06 | 0.311 | 3.05 |

APPENDIX A.1 - EXPERIMENTAL DATA

Table A.1.2. - Equilibrium Concentrations of Nitric Acid in Aqueous and Organic Phases. Organic Phase: 45.1 v% TBP-in-Varsol - Average Values Obtained from determinations at 25, 30 and 35° C.

| AQUEOUS PHASE | | | ORGANIC PHASE | | |
|---|---|---|---|---|---|
| N | d | C | N | d | C |
| 0.048 | 1.000 | 3.03 | 0.0021 | 0.849 | 0.156 |
| 0.091 | 1.001 | 5.76 | 0.0075 | 0.849 | 0.557 |
| 0.173 | 1.003 | 11.0 | 0.020 | 0.850 | 1.48 |
| 0.413 | 1.011 | 26.4 | 0.093 | 0.852 | 6.92 |
| 0.787 | 1.023 | 51.0 | 0.237 | 0.855 | 17.8 |
| 1.49 | 1.043 | 99.0 | 0.49 | 0.862 | 37.2 |
| 1.98 | 1.063 | 133 | 0.65 | 0.866 | 49.7 |
| 2.38 | 1.076 | 162 | 0.77 | 0.869 | 59.2 |
| 3.06 | 1.100 | 212 | 0.94 | 0.874 | 72.8 |

## APPENDIX A.2 - EXPERIMENTAL DATA

Table A.2.1. - Mixed Cartridge. HETS Determinations. Organic Phase Continuous.
Frequency: 102.8 cy/min. Organic/Aqueous Flow Ratio: 10.
Entering Organic Flow: 20 l/hr. TBP Concentration = 45.0 ± 0.1 v%.

| AQUEOUS PHASE | | | | | | | ORGANIC PHASE | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| $a_c$ | $y_{n+1}$ | $d_{n+1}$ | $y_1$ | $d_1$ | $Q_1$ | $e^a$ | $x_o$ | $d_o$ | $x_n$ | $d_n$ | $Q_n$ | $e^o$ | H |
| 1.9 | 0.000 | 0.851 | 0.302 | 0.860 | 30.8 | 0.18 | 2.94 | 1.095 | 0.126 | 1.004 | 3.09 | 0.006 | 1.5 |
| 2.3 | 0.004 | 0.851 | 0.287 | 0.860 | 30.0 | 0.05 | 2.96 | 1.096 | 0.101 | 1.003 | 3.07 | 0 | 3.0 |
| 2.3 | 0.001 | 0.850 | 0.298 | 0.860 | 20.2 | - | 3.00 | 1.098 | 0.097 | 1.003 | 1.84 | - | 1.3 |
| 2.7 | 0.005 | 0.850 | 0.312 | 0.860 | 20.4 | 0.04 | 2.96 | 1.096 | 0.123 | 1.003 | 2.04 | 0.05 | 3.8 |
| 3.1 | 0.005 | 0.850 | 0.297 | 0.860 | 20.2 | 0.1 | 2.96 | 1.096 | 0.135 | 1.005 | 1.90 | 0.03 | 11.9 |
| <u>With Intermediate Settling Zone</u> | | | | | | | | | | | | | |
| 2.3 | 0.001 | 0.850 | 0.311 | 0.859 | 20.3 | 0.02 | 3.01 | 1.098 | 0.118 | 1.003 | 1.94 | 0.04 | 1.8 |
| 2.7 | 0.004 | 0.850 | 0.313 | 0.859 | 20.6 | 0.03 | 3.01 | 1.098 | 0.115 | 1.003 | 2.00 | 0.01 | 3.3 |
| 3.1 | 0.001 | 0.851 | 0.293 | 0.862 | 19.8 | 0.4 | 2.98 | 1.098 | 0.102 | 1.003 | 1.99 | 0.04 | 6.0 |
| 3.5 | 0.001 | 0.851 | 0.306 | 0.861 | 20.1 | 0.3 | 2.98 | 1.098 | 0.127 | 1.004 | 1.93 | 0.05 | 15.8 |

## APPENDIX A.2 - EXPERIMENTAL DATA

Table A.2.2. - Stainless Steel Cartridge. HETS Determinations. Organic Phase Continuous. Frequency: 102.8 cy/min. Organic/Aqueous Flow Ratio: 10. Entering Organic Flow: 20 l/hr. TBP Concentration: 44.9 ± 0,1 v%.

| ORGANIC PHASE | | | | | | | AQUEOUS PHASE | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| $a_c$ | $y_{n+1}$ | $d_{n+1}$ | $y_1$ | $d_1$ | $Q_1$ | $e^a$ | $x_o$ | $d_o$ | $x_n$ | $d_n$ | $Q_n$ | $e^o$ | H |
| 1.9 | 0.001 | 0.853 | 0.308 | 0.862 | 19.6 | 0.02 | 3.01 | 1.098 | 0.117 | 1.004 | 1.95 | 0.03 | 0 |
| 2.3 | 0.001 | 0.853 | 0.296 | 0.862 | 19.9 | 0.01 | 2.98 | 1.096 | 0.099 | 1.003 | 1.95 | 0.03 | 1.0 |
| 2.7 | 0.001 | 0.851 | 0.308 | 0.860 | 20.2 | 0.05 | 2.98 | 1.096 | 0.104 | 1.003 | 1.97 | 0.04 | 2.6 |
| 3.1 | 0.001 | 0.853 | 0.284 | 0.862 | 20.0 | 0.03 | 2.98 | 1.096 | 0.099 | 1.004 | 1.95 | 0.03 | - |
| <u>With Intermediate Settling Zone</u> | | | | | | | | | | | | | |
| 2.3 | 0.000 | 0.853 | 0.325 | 0.862 | 20.0 | 0.02 | 3.00 | 1.097 | 0.134 | 1.004 | 1.98 | 0.03 | 0.8 |
| 2.7 | 0.002 | 0.851 | 0.306 | 0.859 | 20.6 | 0.03 | 3.01 | 1.098 | 0.112 | 1.004 | 1.98 | 0 | 2.1 |
| 3.1 | 0.000 | 0.853 | 0.323 | 0.862 | 19.4 | 0.04 | 3.00 | 1.097 | 0.105 | 1.004 | 1.93 | 0.1 | 3.6 |
| 3.5 | 0.001 | 0.853 | 0.303 | 0.862 | 19.9 | 0.02 | 3.00 | 1.097 | 0.112 | 1.004 | 2.15 | 0.02 | 6.7 |

## APPENDIX A.2 - EXPERIMENTAL DATA

Table A.2.3. - No. 1-Lucite Cartridge. HETS Determinations. Organic Phase Continuous.
Frequency: 102.8 cy/min. Organic/Aqueous Flow Ratio: 10.
Entering Organic Flow: 20 l/hr. TBP Concentration: 45.1 ± 0.1 v%.

| AQUEOUS PHASE | | | | | | | ORGANIC PHASE | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| $a_c$ | $y_{n+1}$ | $d_{n+1}$ | $y_1$ | $d_1$ | $Q_1$ | $e^a$ | $x_o$ | $d_o$ | $x_n$ | $d_n$ | $Q_n$ | $e^o$ | H |
| 1.5 | 0.001 | 0.853 | 0.301 | 0.862 | 19.8 | - | 2.998 | 1.10 | 0.167 | 1.006 | 1.88 | - | - |
| 1.9 | 0.001 | 0.853 | 0.305 | 0.862 | 19.9 | - | 2.998 | 1.10 | 0.120 | 1.004 | 2.03 | - | - |
| 2.3 | 0.002 | 0.853 | 0.304 | 0.858 | 20.3 | 0.02 | 3.007 | 1.10 | 0.126 | 1.004 | 1.99 | 0.04 | 22.8 |
| 2.7 | 0.002 | 0.853 | 0.300 | 0.862 | 14.7 | 0.02 | 3.007 | 1.10 | 0.190 | 1.007 | 1.41 | 0.01 | 27 |
| With Intermediate Settling Zone | | | | | | | | | | | | | |
| 1.9 | 0.001 | 0.853 | 0.323 | 0.863 | 19.7 | 0.01 | 3.014 | 1.10 | 0.154 | 1.005 | 1.92 | 0.02 | - |
| 2.3 | 0.001 | 0.853 | 0.303 | 0.862 | 19.8 | - | 3.014 | 1.10 | 0.126 | 1.004 | 1.95 | - | - |
| 2.3 | 0.001 | 0.854 | 0.299 | 0.862 | 20.2 | 0.03 | 2.998 | 1.10 | 0.122 | 1.003 | 1.88 | 0.03 | - |
| 2.7 | 0.001 | 0.853 | 0.279 | 0.862 | 20.1 | 0.07 | 3.014 | 1.10 | 0.090 | 1.003 | 1.96 | 0.1 | 11.2 |
| 3.1 | 0.001 | 0.853 | 0.304 | 0.864 | 19.9 | 0.04 | 3.014 | 1.10 | 0.120 | 1.001 | 1.91 | 0.1 | 15.0 |

APPENDIX A.2 - EXPERIMENTAL DATA

Table A.2.4. - No. 2-Lucite and Nozzle Plate Cartridges. HETS Determinations. Organic Phase Continuous. Frequency: 102.8 cy/min. Organic/Aqueous Flow Ratio: 10. Entering Organic Flow: 20 l/hr. TBP Concentration: 45,1 ± 0,1 v%.

| ORGANIC PHASE | | | | | | | AQUEOUS PHASE | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| $a_c$ | $y_{n+1}$ | $d_{n+1}$ | $y_1$ | $d_1$ | $Q_1$ | $e^a$ | $x_o$ | $d_o$ | $x_n$ | $d_n$ | $Q_n$ | $e^o$ | H |
| <u>No. 2-Lucite Cartridge</u> | | | | | | | | | | | | | |
| 1.9 | 0.001 | 0.853 | 0.275 | 0.862 | 20.5 | - | 2.979 | 1.10 | 0.164 | 1.006 | 1.9 | - | - |
| 1.9 | 0.001 | 0.853 | 0.269 | 0.863 | 20.3 | - | 2.979 | 1.10 | 0.155 | 1.003 | 1.85 | - | - |
| 2.3 | 0.001 | 0.854 | 0.301 | 0.863 | 20.2 | 0.03 | 3.016 | 1.10 | 0.102 | 1.003 | 1.82 | 0.01 | - |
| 2.7 | 0.001 | 0.853 | 0.290 | 0.862 | 20.2 | 0.05 | 3.016 | 1.10 | 0.115 | 1.004 | 1.8 | 0.01 | 12.7 |
| 3.1 | 0.001 | 0.853 | 0.295 | 0.862 | 18.1 | - | 2.979 | 1.10 | 0.186 | 1.006 | 1.65 | - | - |
| <u>Nozzle Plate Cartridge</u> | | | | | | | | | | | | | |
| 1.1 | 0.001 | 0.853 | 0.304 | 0.863 | 19.8 | 0.02 | 2.949 | 1.10 | 0.321 | 1.011 | 1.94 | 0.02 | - |
| 1.5 | 0.001 | 0.854 | 0.293 | 0.863 | 20.3 | 0.05 | 2.949 | 1.10 | 0.172 | 1.006 | 1.84 | 0.005 | - |
| 1.9 | 0.000 | 0.853 | 0.293 | 0.863 | 20.2 | 0.06 | 2.949 | 1.10 | 0.186 | 1.006 | 1.90 | 0.03 | 3.5 |
| 2.3(*) | 0.001 | 0.854 | 0.291 | 0.863 | 20.4 | 0.16 | 3.016 | 1.10 | 0.240 | 1.008 | 1.96 | 0.01 | 21.8 |
| 2.3 | 0.001 | 0.854 | 0.285 | 0.863 | 20.1 | 0.2 | 3.016 | 1.10 | 0.226 | 1.008 | 1.86 | 0.01 | 7.6 |

(*) Nozzles pointing downward. All other points with nozzles pointing upward.

## APPENDIX A.3 - EXPERIMENTAL DATA

Table A.3.1. = Flooding Capacity at 25° C (1 $cm^{-2} hr^{-1}$). Frequency: 102.8 cy/min.
Organic/Aqueous Flow Ratio: 10. Aqueous Phase: 3 ± 0.02 N $HNO_3$.
Organic Phase: 45 ± 0.2 v%. TBP-in-Varsol.
Column Cross-Sectional Area: 25 $cm^2$.

| Cartridge | Amplitude x Frequency ($cm\ sec^{-1}$) | | | | | |
|---|---|---|---|---|---|---|
| | 2.60 | 3.34 | 3.87 | 4.62 | 5.29 | 5.99 |
| Mixed (complete) | - | 3.30 | 2.07 | 1.25 | 0.93 | 0.84 |
| | | 3.13 | 2.20 | 1.19 | 0.97 | - |
| Mixed (with ISZ) | - | 2.86 | 2.24 | 1.76 | 1.32 | 1.14 |
| | | 3.08 | 2.55 | 1.59 | 1.32 | 1.10 |
| | | - | - | 1.63 | - | - |
| Stainless Steel (complete) | - | - | ~3.96 | 1.63 | 1.23 | 0.97 |
| Stainless Steel (with ISZ) | - | - | 3.53 | 1.65 | 1.32 | 1.19 |
| | | - | 3.53 | 1.63 | 1.23 | 1.10 |
| No. 1-Lucite (complete) | - | 2.12 | 1.5 | 1.06 | 0.75 | - |
| No. 1-Lucite (with ISZ) | - | 2.91 | 2.07 | 1.37 | 0.97 | - |
| No. 2-Lucite (complete) | - | 2.78 | 1.81 | 1.32 | 1.10 | 0.88 |
| Nozzle-Plate (complete) | 2.90 | 1.56 | 1.19 | - | - | - |

## APPENDIX A.3 - EXPERIMENTAL DATA

Table A.3.2. - Flooding Capacity as Function of Temperature (1 $cm^{-2}hr^{-1}$).
Frequency: 102.8 cy/min. Organic/Aqueous Flow Ratio: 10.
Aqueous Phase: 3 ± 0.02 N $HNO_3$. Organic Phase: 45 ± 0.2 v%.
TBP-in-Varsol. Column Cross-Sectional Area: 25 $cm^2$.

| | CARTRIDGE | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| af | Mixed | | | Stainless Steel | | | No. 1-Lucite | | |
| ($cm\ sec^{-1}$) | 22°C | 25°C | 30°C | 22°C | 25°C | 30°C | 20°C | 25°C | 30°C |
| 2.60 | - | - | - | - | - | - | 3.08 | - | - |
| 3.34 | 3.08 | 3.30 | 3.39 | - | - | - | 1.85 | 2.11 | 2.64 |
| 3.87 | 1.98 | 2.07 | 2.25 | 3.21 | ~3.96 | - | 1.32 | 1.50 | 1.98 |
| 4.62 | 1.23 | 1.25 | 1.28 | 1.54 | 1.63 | 1.98 | 0.92 | 1.06 | 1.27 |
| 5.29 | 0.92 | - | 0.93 | 1.14 | 1.23 | 1.28 | - | 0.75 | 0.88 |
| 5.99 | 0.84 | - | 0.84 | 0.92 | 0.97 | 0.97 | - | - | - |

## APPENDIX B.1 - CALCULATED DATA

Table B.1.1. - Mixed Cartridge. Organic Phase Continuous. Frequency: 102.8 cy/min. Organic/Aqueous Flow Ratio: 10. Entering Organic Flow: 20 l/hr. TBP Concentration: 45 ± 0.1 v%. Height of Contact Section: 114.5 cm.

| af ($cm\ sec^{-1}$) | Number of Stages | HETS (cm) | L/V (g/g) |
|---|---|---|---|
| 3.34 | 3.7 | 31 | 0.115 |
| 3.87 | 4.9 | 23 | 0.106 |
| 3.87 | 4.6 | 25 | 0.110 |
| 4.62 | 4.5 | 25 | 0.116 |
| 5.29 | 3.9 | 28 | 0.113 |
| <u>With Intermediate Settling Zone</u> | | | |
| 3.87 | 4.0 | 29 | 0.116 |
| 4.62 | 4.6 | 25 | 0.115 |
| 5.29 | 4.2 | 27 | 0.109 |
| 5.99 | 3.9 | 29 | 0.115 |

## APPENDIX B.1 - CALCULATED DATA

Table B.1.2. - Stainless Steel Cartridge. Organic Phase Continuous.
Frequency: 102.8 cy/min. Organic/Aqueous Flow Ratio: 10.
Entering Organic Flow: 20 l/hr. TBP Concentration: 44.9 ± 0.1 v
Height of Contact Section: 113.2 cm.

| af ($cm\ sec^{-1}$) | Number of Stages | HETS (cm) | L/V (g/g) |
|---|---|---|---|
| 3.34 | 4.0 | 28 | 0.115 |
| 3.87 | 4.3 | 26 | 0.110 |
| 4.62 | 4.5 | 25 | 0.114 |
| 5.29 | 4.3 | 26 | 0.105 |
| <u>With Intermediate Settling Zone</u> | | | |
| 3.87 | 3.8 | 30 | 0.122 |
| 4.62 | 4.3 | 26 | 0.113 |
| 5.29 | 4.5 | 25 | 0.120 |
| 5.99 | 4.0 | 28 | 0.113 |

## APPENDIX B.1 - CALCULATED DATA

Table B.1.3. - No. 1-Lucite Cartridge. Organic Phase Continuous.
Frequency: 102.8 cy/min. Organic/Aqueous Flow Ratio: 10.
Entering Organic Flow: 20 l/hr. TBP Concentration: 45.1 ± 0.1 v/
Height of Contact Section: 116.5 cm.

| af ($cm\ sec^{-1}$) | Number of Stages | HETS (cm) | L/V (g/g) |
|---|---|---|---|
| 2.60 | 3.1 | 38 | 0.113 |
| 3.34 | 3.9 | 30 | 0.113 |
| 3.87 | 3.8 | 31 | 0.114 |
| 4.62 | 2.9 | 40 | 0.115 |
| <u>With Intermediate Settling Zone</u> | | | |
| 3.34 | 3.5 | 33 | 0.121 |
| 3.87 | 3.8 | 31 | 0.113 |
| 3.87 | 3.8 | 31 | 0.113 |
| 4.62 | 4.4 | 27 | 0.101 |
| 5.29 | 3.8 | 31 | 0.112 |

## APPENDIX B.1 - CALCULATED DATA

Table B.1.4. - No. 2-Lucite and Nozzle-Plate Cartridges. Organic Phase Continuous. Frequency: 102.8 cy/min. Organic/Aqueous Flow Ratio: 10. Entering Organic Flow: 20 l/hr. TBP Concentration: 45.1 ± 0.1 v%.

| $a_f$ (cm $sec^{-1}$) | Number of Stages | HETS (cm) | L/V (g/g) |
|---|---|---|---|
| **No. 2-Lucite** (height of contact section: 117.2 cm) | | | |
| 3.34 | 3.0 | 39 | 0.101 |
| 3.34 | 3.0 | 39 | 0.104 |
| 3.87 | 4.3 | 27 | 0.110 |
| 4.62 | 3.9 | 30 | 0.107 |
| 5.29 | 2.9 | 40 | 0.113 |
| **Nozzle-Plate** (height of contact section: 113.5 cm) | | | |
| 1.90 | 2.1 | 54 | 0.123 |
| 2.60 | 3.0 | 38 | 0.113 |
| 3.34 | 2.9 | 39 | 0.113 |
| 3.87 | 2.5 | 45 | 0.109 |
| 3.87(*) | 2.5 | 45 | 0.111 |

(*) Nozzles pointing downward. All other points with nozzles pointed upward.

## APPENDIX C

### Sample Calculation.

Let us consider an experimental run with the stainless steel cartridge, for example at an amplitude of 1.9 cm in the column. Thus, from Table A.2.2., we have:

$a_c$ = 1.9 cm
$f$ = 102.8 cy/min = 1.71 cy/sec
$af$ = 3.34 cm/sec
$x_o$ = 3.01 moles/liter (d = 1.098 g/ml)
$y_1$ = 0.308 moles/liter (d = 0.862 g/ml)
$x_n$ = 0.117 moles/liter (d = 1.004 g/ml)
$y_{n+1}$ = 0.001 moles/liter (d = 0.853 g/ml)
$h$ = 113.2 cm

Neglecting entrainment and miscibility of solvents, we can find the concentrations of the different streams in grams of $HNO_3$ per gram of $HNO_3$-free solvent, by means of the formula:

$$C = (63N)/(1000d - 63N)$$
$$= (1)/(15.9\frac{d}{N} - 1) \quad g\text{-}HNO_3/g\text{-}HNO_3\text{-free solvent}$$

Thus,

$x_o$ = $206.5 \times 10^{-3}$ g-$HNO_3$/g water
$y_1$ = $23.0 \times 10^{-3}$ g-$HNO_3$/g TBP-Varsol
$x_n$ = $7.40 \times 10^{-3}$ g-$HNO_3$/g water
$y_{n+1}$ = $0.06 \times 10^{-3}$ g-$HNO_3$/g TBP-Varsol

The equilibrium concentrations can now be plotted in the equilibrium diagram and the operating line drawn (Fig.C.1). The number of ideal extraction stages is then found by the usual stepping procedure of McCabe-Thiele. Therefore,

number of extraction stages = 4
height of contacting section = 113.2 cm
HETS = 113.2/4 = 28.3 cm $\simeq$ 28 cm

which is the value reported in Table B.2.1.

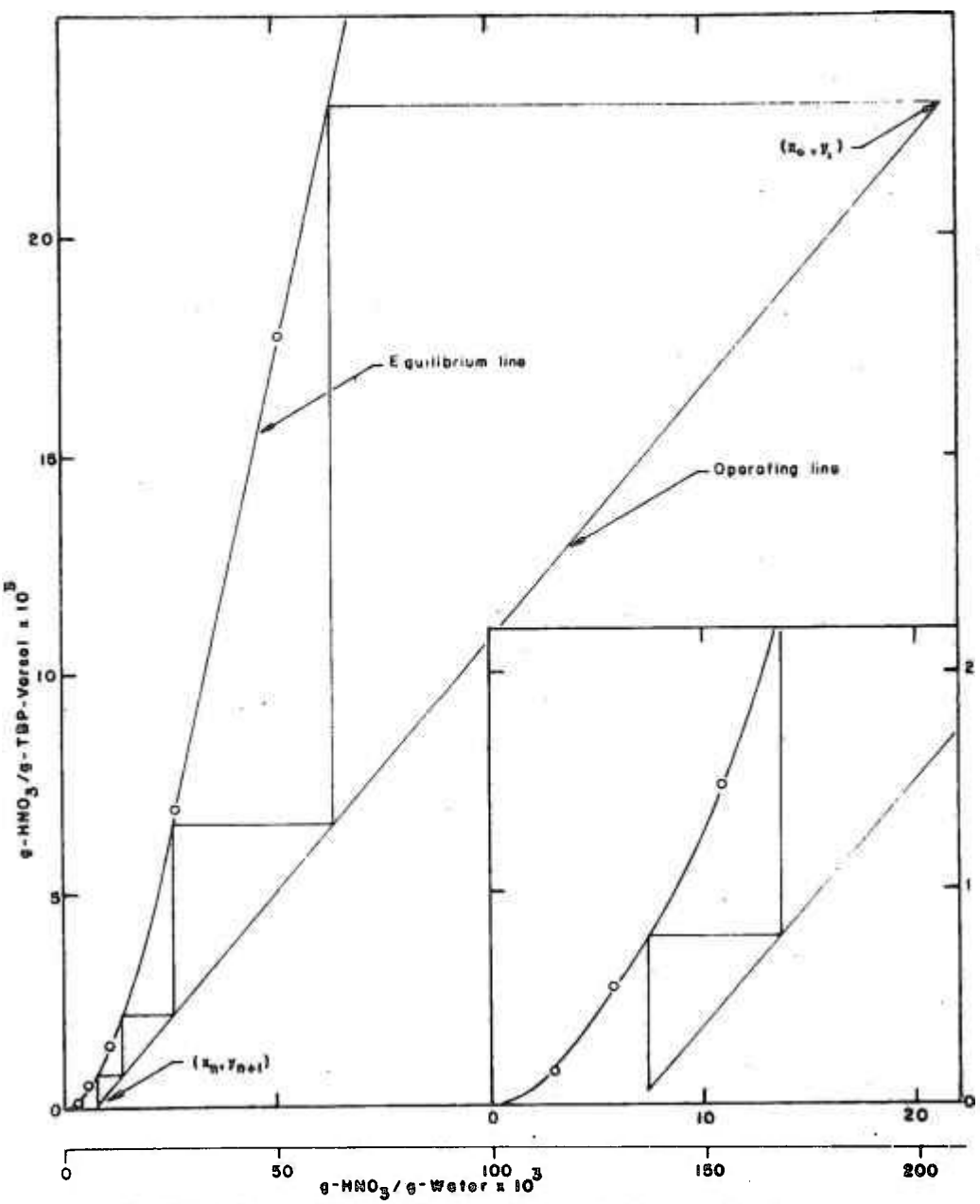


Fig. C-1 - Graphical Determination of Number of Ideal Extraction Stages. McCabe-Thiele Method.

The actual aqueous to organic flow ratio in the column is given by

$$\frac{L}{V} = (y_1 - y_{n+1})/(x_o - x_n)$$

$$= (23.0 - 0.06)/(206.5 - 7.4)$$

$$= 0.115 \text{ g water/g TBP-Varsol}$$

which is the slope of the operating line.

## 6. NOMENCLATURE

### Symbols:

| | |
|---|---|
| $a$ | total amplitude of motion, cm |
| $a_c$ | total amplitude of motion in the column, cm |
| af | amplitude x frequency product, cm/sec |
| C | nitric acid concentration, g $HNO_3$/g $HNO_3$-free solvent |
| d | density at 25° C, g/cm$^3$ |
| $d_o$ | density at 25° C, of aqueous feed, g/cm$^3$ |
| $d_1$ | density at 25° C, of extract phase at equilibrium, g/cm$^3$ |
| $d_n$ | density at 25° C, of raffinate at equilibrium, g/cm$^3$ |
| $d_{n+1}$ | density at 25° C, of organic feed, g/cm$^3$ |
| $e^a$ | entrainment of aqueous phase in extract, v% |
| $e^o$ | entrainment of organic phase in raffinate, v% |
| f | frequency of cyclic motion, cy/sec |
| h | height of contacting section, cm |
| H | hold-up of the dispersed phase in the column at equilibrium, expressed in centimeters height. |
| HETS | height equivalent to a theoretical stage, cm |
| ISZ | intermediate settling zone |
| K | distribution coefficient of nitric acid |
| L/V | aqueous to organic flow ratio, g water/g TBP-Varsol |
| N | normality of nitric acid |
| $N^a$ | normality of nitric acid in aqueous phase at equilibrium |
| $N^o$ | normality of nitric acid in organic phase at equilibrium |
| $P_f$ | performance factor, hr$^{-1}$ |
| $Q_1$ | flow rate of extract phase at equilibrium, l/hr |
| $Q_n$ | flow rate of raffinate at equilibrium, l/hr |
| $x_o$ | nitric acid concentration of aqueous feed, moles/l |
| $x_n$ | nitric acid concentration of raffinate at equil., moles/l |
| $y_1$ | nitric acid concentration of extract at equil., moles/l |
| $y_{n+1}$ | nitric acid concentration of organic feed, moles/l |